

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

CHEMISTRY 9701/32

Paper 32 Advanced Practical Skills

May/June 2009

CONFIDENTIAL INSTRUCTIONS

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



The Supervisor's attention is drawn to the form on page 11 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.

This document consists of 9 printed pages and 3 blank pages.



Safety

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 'Apparatus' on the use of pipette fillers, safety goggles and plastic gloves.

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 N = dangerous for the environment

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.

Before the Examination

1 Access to the question paper is NOT permitted in advance of the examination.

2 Preparation of materials

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.

All solutions should be bulked and mixed thoroughly before use to ensure uniformity.

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

Supervisors are asked to carry out any confirmatory tests given on pages 4, 5 and 6 to ensure the materials supplied are appropriate.

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

3 Labelling of materials

Materials must be labelled as specified in these instructions. Materials with an **FB** code number should be so labelled **without** the identities being included on the label. Where appropriate the identity of an **FB** coded chemical is given in the question paper itself.

4 Identity of materials

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

5 Size of group

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.

Apparatus

- 1 In addition to the fittings ordinarily contained in a chemical laboratory, the apparatus and materials specified below will be necessary.
- 2 Pipette fillers (or equivalent safety devices), safety goggles and disposable plastic gloves should be used where necessary.
- 3 For each candidate
 - $2 \times 50 \, \text{cm}^3$ burettes
 - 2 × burette clamps
 - $2 \times stands$
 - $2 \times \text{funnels}$ for filling burettes
 - $1 \times 250 \, \text{cm}^3$ graduated (volumetric) flask, labelled **FB 3**
 - $1 \times 250 \, \text{cm}^3$ conical flask
 - $1 \times 25 \, \text{cm}^3$ pipette
 - 1 × pipette filler
 - 1 × white tile
 - 1×-10 °C to 110 °C by 1 °C thermometer
 - $1 \times$ wash bottle containing distilled water
 - 1 × tripod and gauze
 - 1 × Bunsen burner
 - 1 × heat proof mat
 - 1 × boiling-tube, labelled NaHCO₃
 - $1 \times 250 \, \text{cm}^3 \, \text{beaker}$
 - 1 × foamed plastic (polystyrene) cup. The cup should be of sufficient capacity to prevent loss of liquid when $8g \, \text{Na}_2 \text{CO}_3$ is added to $50 \, \text{cm}^3$ of $3 \, \text{mol dm}^{-3} \, \text{HCl}$
 - $1 \times$ measuring cylinder, to measure $50 \, \text{cm}^3$
 - $1 \times \text{test-tube holder}$
 - $12 \times \text{test-tubes}$
 - 2 × rubber bungs to fit test-tubes
 - $1 \times \text{test-tube rack}$
 - 1 × marker pen or blank adhesive labels to label test-tubes
 - 2 × teat/squeeze pipettes
 - 1 × spatula

access to a balance weighing to 0.1 g or better

Where balance access is restricted, some candidates should be instructed to commence the examination on each of the questions.

The laboratory should be well ventilated during the examination to prevent a build up of unpleasant fumes.

Chemicals Required

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 Particular requirements

hazard	label	per candidate	identity	notes (hazards given in this column are for the raw materials)
	FB 1	150 cm ³	$0.023 \text{mol dm}^{-3} \text{potassium}$ $\text{manganate}(VII)$	Dissolve 3.63g of KMnO $_4$ in each dm 3 of distilled water.
Ξ	FB 2	70 cm ³	0.35 mol dm ⁻³ ethanedioic acid	Dissolve 44.1 g of $H_2C_2O_4.2H_2O$ [H] in each dm ³ of distilled water.
Ξ	1 mol dm ⁻³ sulfuric acid	150 cm ³	1 mol dm ⁻³ sulfuric acid	Cautiously pour $55\mathrm{cm^3}$ of concentrated (98%) sulfuric acid [C] into $500\mathrm{cm^3}$ of distilled water with continuous stirring. Make the solution up to $1\mathrm{dm^3}$ with distilled water. <i>Care:</i> Concentrated H_2SO_4 is very corrosive.
	distilled water	500 cm ³	distilled water	
Check Titre Pipette 10.0 reaches a te Adjust the o	Check Titre Pipette 10.0 cm ³ of FB 2 into a conical flask and add 25 cm ³ reaches a temperature of 75 °C. Titrate with FB 1 until the fi Adjust the concentration of FB 1, if necessary, to give a	a conical fl °C. Titrate f FB 1, if ne	lask and add 25 cm³ of 1.0 me with FB 1 until the first perme ecessary, to give a titre of 6	Check Titre Pipette 10.0 cm ³ of FB 2 into a conical flask and add 25 cm ³ of 1.0 mol dm ⁻³ sulfuric acid and 40 cm ³ distilled water. Warm the flask until the solution reaches a temperature of 75 °C. Titrate with FB 1 until the first permanent pale pink colour is formed. Adjust the concentration of FB 1, if necessary, to give a titre of 60.75 ± 0.25 cm ³ .
[I]	FB 4	6.0 – 7.0 g	anhydrous sodium carbonate	Between 6.0 and 7.0g of powdered Na ₂ CO ₃ [H] – in a stoppered boiling-tube, labelled FB 4 . Anhydrous Na ₂ CO ₃ from the bottle should be heated for an hour at 130 °C in an oven, then allowed to cool in a desiccator.
	FB 5	15g	sodium hydrogencarbonate	About 15g of NaHCO ₃ – in a stoppered container.
[H]	3mol dm ⁻³ hydrochloric acid	150 cm³	3 mol dm ⁻³ hydrochloric acid	Dilute 258 cm 3 of concentrated (35% w/w; approximately 11 mol dm $^{-3}$) acid [C] to 1 dm 3 .
[T][N]	FB 6	1 g	sodium bromide	About 1 g of NaBr (or KBr) – in a stoppered container.
[N][L]	FB 7	1 g	sodium iodide	About 1 g of NaI (or KI) – in a stoppered container.
[H][N]	FB 8	10 cm ³	0.10 mol dm ⁻³ zinc sulfate	Dissolve 28.8g of $ZnSO_4.7H_2O$ [H][N] in each dm^3 of solution.
	FB 9	10 cm ³	0.10 moldm ⁻³ magnesium sulfate	Dissolve 24.6g of $\mathrm{MgSO_{4.7H_2}O}$ in each $\mathrm{dm^3}$ of solution.

Particular requirements continued

hazard 6005 SET	label	per candidate	identity	notes (hazards given in this column are for the raw materials)
<u></u>	concentrated sulfuric acid	5cm³	concentrated sulfuric acid	Fresh, concentrated sulfuric acid [C] – provided in a dropping bottle or with a pipette. The container should be clearly marked to indicate the corrosive nature of the acid.
	starch solution	10cm³	2% starch solution	Mix 2g of soluble starch to a paste with a little distilled water, taken from $100\mathrm{cm}^3$, of water. Boil the remaining water and pour the paste into the boiling water. Stir and cool.
NH	aqueous bromine (bromine water)	2cm³	0.1% (v/v) aqueous bromine	Dissolve 1.0 cm ³ of bromine [T][C][N] in each 1000 cm ³ of solution. Provide in a stoppered tube. Safety goggles and plastic gloves should be used when making up the solution for candidates. 1 mol dm ⁻³ sodium thiosulfate should be available to treat any spillage. Freshly purchased bromine water would be a suitable alternative.

N.B. Small amounts of unpleasant gas will be produced in question 3. The laboratory should be well ventilated.

The standard bench 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. ო

hazard	label	identity	notes (hazards given in this column are for the raw materials)
Ξ	dilute hydrochloric acid	2.0 moldm ⁻³ HC l	Dilute 172 cm 3 of concentrated (35% w/w; approximately 11 mol dm $^{-3}$) acid [C] to 1 dm 3 .
ට	aqueous sodium hydroxide	2.0 moldm ⁻³ NaOH	Dissolve 80.0g of NaOH [C] in each dm ³ of solution. Care – the process of solution is exothermic and any concentrated solution is very corrosive.
Ξ	aqueous ammonia	2.0 moldm ⁻³ NH ₃	Dilute 112 cm ³ of concentrated (35% w/w) ammonia [C][N] to 1 dm ³ .
Ε	0.1 mol dm ⁻³ barium chloride	0.1 moldm ⁻³ barium chloride	$0.1 \mathrm{moldm^{-3}}$ barium chloride Dissolve 24.4g of BaC I_2 .2H ₂ O [T] in each dm ³ of solution.
Ξ	or 0.1 mol dm ⁻³ barium nitrate	or 0.1 mol dm ⁻³ barium nitrate	Dissolve 26.1 g of Ba(NO_3) ₂ [H] in each dm ³ of solution.
	0.1 mol dm ⁻³ lead(II) nitrate	$0.1\mathrm{moldm^{-3}}$ lead(II) nitrate	Dissolve 33.1 g of $Pb(NO_3)_2$ [T][O][N] in each dm^3 of solution.
[H]	0.05 mol dm ⁻³ silver nitrate	0.05 mol dm ⁻³ silver nitrate	Dissolve 8.5g of $AgNO_3$ [C][N] in each dm^3 of solution.

The reagents, materials and apparatus to test the gases listed in the syllabus must be available to candidates. 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.

hazard	label	identity	notes (hazards given in this column are for the raw materials)
Ξ	limewater	saturated aqueous calcium hydroxide, Ca(OH) ₂	Prepare fresh limewater by leaving distilled water to stand over solid calcium hydroxide, [H], for several days, shaking occasionally. Decant or filter the solution.
[N][L]	aqueous potassium dichromate(${ m VI})$	0.05 mol dm ⁻³ K ₂ Cr ₂ O ₇ 0.05 mol dm ⁻³ H ₂ SO ₄	 D.05 moldm⁻³ K₂Cr₂O₇ Dissolve 14.8g of K₂Cr₂O₇ [T][N] in 50.0 cm³ of 1.0 moldm⁻³ dilute sulfuric acid [H]. Make the solution up to 1 dm³ with distilled water. D.05 moldm⁻³ H₂SO₄ The use of plastic gloves may be considered to prevent contact with skin.

red and blue litmus paper, plain filter paper strips for use with aqueous potassium dichromate(VI), aluminium foil for testing for nitrate/nitrite, wooden splints, the apparatus normally used in the Centre for use with limewater in testing for carbon dioxide

Responsibilities of the Supervisor during the Examination

1 The Supervisor, or other competent chemist must carry out the experiments in question 1 and question 2 and complete tables 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.

2 The Supervisor must complete the Report Form on page 11 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.

The Supervisor must give details on page 12 of any particular difficulties experienced by a candidate, especially if the Examiner would be unable to discover this from the written answers.

After the Examination

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 11 and 12).
- 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.

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.

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REPORT FORM

This form must be	completed a	nd sent to the	Examiner in the	e envelone wit	h the scripts.
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Cer	itre N	Number!	Name of Centre				
1	Sup	pervisor's Results					
	the		Question 1 and Question 2 on a spare copy of Results' and showing the Centre number and				
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	ng preparation;				
	(b)	difficulties due to faulty apparatus or materia	als;				
	(c)	accidents to apparatus or materials;					
	(d)	assistance with respect to colour-blindness.					

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.

normal 'Application for Special Consideration' form.

Other cases of hardship, e.g. illness, temporary disability, should be reported direct to CIE on the



publisher will be pleased to make amends at the earliest possible opportunity.