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

CANDIDATE NAME	

CENTRE NUMBER		CANDIDATE NUMBER	

4991551712

CHEMISTRY **9701/36**

Advanced Practical Skills **October/November 2010**

2 hours

Candidates answer on the Question Paper.

Additional Materials: As listed in the 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.
DO **NOT** WRITE IN ANY BARCODES.

Answer **all** questions.
You may lose marks if you do not show your working or if you do not use appropriate units.
Use of a Data Booklet is unnecessary.

Qualitative Analysis Notes are printed on pages 11 and 12.

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	
3	
Total	

This document consists of **12** printed pages.

DC (LEO) 28255/3

UNIVERSITY *of* CAMBRIDGE International Examinations

You must prepare Flask A and Flask B in Question 2 before starting Question 1. Shake each flask periodically during the time you spend on Question 1.

For Examiner's Use

1 **FB 1** is 0.125 mol dm^{-3} sulfuric acid, H_2SO_4.
FB 2 is an aqueous solution of sodium hydroxide, NaOH.

You are to determine the concentration, in mol dm^{-3}, of the sodium hydroxide in **FB 2**.

(a) Method

- Fill a burette with **FB 1**.
- Run between 45.50 cm^3 and 46.50 cm^3 of **FB 1** from the burette into the 250 cm^3 graduated (volumetric) flask, labelled **FB 3**.
- Make up to the mark with distilled water.
- Shake the flask to mix the solution.

In the space below record your burette readings and the volume of **FB 1** added to the graduated flask.

You are reminded to shake Flask A and Flask B periodically.

Titration

- Fill a second burette with **FB 2**.
- Pipette 25.0 cm^3 of **FB 3**, the diluted acid, into a conical flask.
- Add to the flask a few drops of phenolphthalein indicator.
- Place the flask on a white tile.
- Titrate the acid in the flask with **FB 2**.
  At the end-point a “permanent” pink colour will remain in the solution.
- **Note**: The “permanent” pink colour will fade over several minutes as carbon dioxide is absorbed from the atmosphere.

You should perform a **rough titration**.
In the space below record your burette readings for this rough titration.

The rough titre is .... cm^3.

- Carry out as many accurate titrations as you think necessary to obtain consistent results
- Record in a suitable form on page 3 all of your burette readings and the volume of **FB 2** added in each accurate titration.
- Make certain any recorded results show the precision of your practical work.

You will require the burette containing FB 2 for Question 2.

For Examiner's Use

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VII	

[7]

(b) From your accurate titration results obtain a suitable value to be used in your calculation. Show clearly how you have obtained this value.

25.0 cm^3 of **FB 3** required .... cm^3 of **FB 2**.
[1]

Calculations
Show your working and appropriate significant figures in the final answer to **each** step of your calculations.

(c) **(i)** Calculate how many moles of H_2SO_4 in **FB 1** were run from the burette into the 250 cm^3 graduated, (volumetric) flask.

.... mol of H_2SO_4 were run from the burette into the graduated flask.

(ii) Calculate how many moles of H_2SO_4 in **FB 3** were pipetted from the graduated flask into the conical flask in each titration.

.... mol of H_2SO_4 were pipetted into the conical flask.

(iii) Calculate how many moles of NaOH reacted with the H_2SO_4 in **(ii)**.

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

The H_2SO_4 in the titration flask reacted with .... mol of NaOH.

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(iv) Calculate the concentration, in mol dm^{-3}, of NaOH in **FB 2**.

The concentration of NaOH in **FB 2** is ………………… mol dm^{-3}.
[5]

(d) The individual error in any burette reading is ±0.05 cm^3.
Two students, A and B, record identical burette readings.

final burette reading	25.60 cm^3
initial burette reading	1.35 cm^3
volume added	24.25 cm^3

Explain the following.

(i) The initial burette reading made by student A was 0.05 cm^3 greater than the true value but the volume added was exactly 24.25 cm^3.

....

....

(ii) The initial burette reading made by student B was 0.05 cm^3 less than the true value and the actual volume added was exactly 24.15 cm^3.

....

....
[2]

(e) In the instructions for the experiment you were told that the "permanent" pink colour at the end-point would fade over a few minutes as carbon dioxide is absorbed from the atmosphere.

(i) Explain why absorption of carbon dioxide at the end-point would reverse the indicator colour change seen in the titration.

....

....

(ii) Suggest a modification to the titration method, using the same indicator, that would overcome this problem.

....

....
[2]

[Total: 17]

2 **FB 4** is 0.050 mol dm^{-3} sodium hydroxide solution.
FB 5 is 0.200 mol dm^{-3} propanoic acid, $C_2H_5CO_2H$.
FB 6 is an organic liquid that does not mix with water.

For Examiner's Use

Propanoic acid dissolves both in water and in the organic layer, **FB 6**.
When an aqueous solution of the acid is shaken with **FB 6**, some of the acid transfers to the organic layer.
The amount of acid remaining in the aqueous layer can be determined by titration with aqueous sodium hydroxide.

Preparation of the mixture in Flask A and in Flask B.

Flask A

- Use a measuring cylinder to place 50 cm^3 of **FB 5** into the stoppered flask labelled **Flask A**.
- Use a second measuring cylinder to add to the flask 40 cm^3 of **FB 6**, the organic liquid.
- Replace the stopper in the flask.

Flask B

- Use the first measuring cylinder to place 50 cm^3 of **FB 5** into the stoppered flask labelled **Flask B**.
- Use the second measuring cylinder to add to the flask 60 cm^3 of **FB 6**, the organic liquid.
- Replace the stopper in the flask.

- Shake both flasks vigorously for about 1 minute.
- **Leave the flasks on the workbench and start Question 1.**
- Shake the flasks for a further minute at intervals during the course of your work on another question.

(a) Titrations

For each flask follow the same procedure.

- Empty the burette containing **FB 2**.
- Rinse the burette thoroughly with **FB 4**.
- Fill the burette with **FB 4**.
- Ensure the two layers have separated – this should take no longer than 1 minute after shaking the flask.
- Pipette 10.0 cm^3 of the **lower** (aqueous) layer into a conical flask. Attach the pipette filler to the pipette before inserting it into the mixture, in order to close the top of the pipette to prevent any of the top (organic) layer from entering the pipette.
- Replace the stopper in the flask.
- Titrate the acid in the conical flask with **FB 4**, using phenolphthalein indicator, as in Question 1.
- **One titration will be sufficient for each experiment but take care to ensure that no errors are made during the procedure**.

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For Examiner's Use

Results
Record, in a single table below, the burette readings and volume of **FB 4** added, for each of **Flask A** and **Flask B**.

[4]

(b) Calculations

In these calculations make use of the following.

- The concentration of NaOH in **FB 4** is 0.050 mol dm^{-3}.
- 50 cm^3 of 0.200 mol dm^{-3} propanoic acid, the volume of acid added to each flask, contains 0.010 mol $C_2H_5CO_2H$.
- 1 mol $C_2H_5CO_2H$ reacts with 1 mol NaOH.

(i) Calculate the volume of **FB 4** that contains 0.010 mol NaOH.
This is the volume of **FB 4** that would have reacted with the propanoic acid in the 50 cm^3 of the aqueous layer, **before** shaking with the organic liquid.

Volume of **FB 4** = ... cm^3

(ii) For each flask, use your titration result in **(a)** to calculate the volume of **FB 4** needed to react with the acid remaining in **50 cm^3** of the aqueous layer, **after** shaking with the organic liquid.

Flask A	**Flask B**
volume of **FB 4** = ... cm^3	volume of **FB 4** = ... cm^3

(iii) The amount of propanoic acid transferred to the organic layer can be represented by the following.

(answer to **(i)** – answer to **(ii)**)

For each flask evaluate this expression.

Flask A (answer to **(i)** – answer to **(ii)**) = ... cm^3

Flask B (answer to **(i)** – answer to **(ii)**) = ... cm^3

[2]

For Examiner's Use

(c) In which flask was most propanoic acid transferred to the organic layer?

Justify your answer.

....

....

....
[1]

(d) It is suggested that shaking the mixture leads to the following equilibrium being established.

$$C_2H_5CO_2H(aq) \rightleftharpoons C_2H_5CO_2H(org)$$

Determine the equilibrium constant by evaluating the expressions in the following table.

(i) Determine the equilibrium constant by evaluating the expressions in the following table. **Ignore units**.

Flask A	**Flask B**
$K_c = \dfrac{\text{answer } \textbf{(b)(iii)}}{\text{answer } \textbf{(b)(ii)}} \times 1.25$	$K_c = \dfrac{\text{answer } \textbf{(b)(iii)}}{\text{answer } \textbf{(b)(ii)}} \times 0.83$
K_c = ...	K_c = ...

(ii) Explain whether or not your results support the idea that equilibrium has been established in each flask.

....

....

....
[1]

[Total: 8]

For Examiner's Use

3 **FB 7**, **FB 8** and **FB 9** are aqueous solutions, each containing cations and anions from those listed on pages 11 and 12 in the Qualitative Analysis Notes.

At each stage of any test you are to record details of the following.

- colour changes seen
- the formation of any precipitate
- the solubility of such precipitates in an excess of the reagent added

Where gases are released they should be identified by a test, **described in the appropriate place 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.
If any solution is warmed, a boiling-tube MUST be used.
Rinse and reuse test-tubes and boiling-tubes where possible.

(a) **(i)** One or more of the solutions **FB 7**, **FB 8** and **FB 9** are believed to contain the ammonium ion, NH_4^+.

Suggest a reagent that would enable you to identify the presence of NH_4^+ and describe how you would use the reagent in an appropriate test.

reagent ....

test ....

....

Use this reagent to test each of the solutions. Record your observations in the table below.

solution	*observation*
FB 7	
FB 8	
FB 9	

I	
II	

(ii) One or more of the solutions contains the sulfate ion, SO_4^{2-}.
Select reagents that would enable you to identify the presence of SO_4^{2-}.
Show clearly, by describing how the reagents will be used, how you would distinguish SO_4^{2-} from the sulfite ion, SO_3^{2-}.

For Examiner's Use

reagents ....

test ....

Use these reagents to test each of the solutions. Record your observations in the table below.

solution	*observation*
FB 7	
FB 8	
FB 9	

III	
IV	
V	

(iii) **Conclusions**

The ammonium ion, NH_4^+, is present in ....

The sulfate ion, SO_4^{2-}, is present in ....

[5]

(b) Use aqueous sodium hydroxide and aqueous ammonia in separate tests to identify any cation (apart from NH_4^+) present in **FB 7**, **FB 8** and **FB 9**.

Present your results for each of the solutions in a suitable form below.

I	
II	
III	
IV	

[4]

(c) Conclusion

For Examiner's Use

Complete the following table.
Place a cross in any box where no cation has been identified.

solution	*cation*	*supporting evidence*
FB 7		
FB 8		
FB 9		

[1]

(d) Carry out the following tests on **FB 10**.
Observe carefully at each stage and record all of your observations in the table.

	test	*observations*
(i)	Place 2 spatula measures of **FB 10** in a dry, hard glass boiling-tube. Heat the solid gently at first, then strongly until no further change is seen. Retain the solid for use in **(ii)**.	
(ii)	Tip the contents of the tube in **(i)** into a second boiling-tube. Add 2 cm depth of dilute hydrochloric acid **a little at a time**. Warm the tube and leave to stand.	

I	
II	
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V	

[5]

[Total: 15]

Qualitative Analysis Notes

Key: [ppt. = precipitate]

1 Reactions of aqueous cations

ion	*reaction with*	
	$NaOH(aq)$	$NH_3(aq)$
aluminium, $Al^{3+}(aq)$	white ppt. soluble in excess	white ppt. insoluble in excess
ammonium, $NH_4^+(aq)$	no ppt. 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 giving dark blue solution
iron(II), $Fe^{2+}(aq)$	green ppt. turning brown on contact with air insoluble in excess	green ppt. turning brown on contact with air 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. rapidly turning brown on contact with air insoluble in excess	off-white ppt. rapidly turning brown on contact with air 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 A*l* foil
nitrite, NO_2^- (aq)	NH_3 liberated on heating with OH^-(aq) and A*l* foil, NO liberated by dilute acids (colourless NO → (pale) brown NO_2 in air)
sulfate, SO_4^{2-} (aq)	gives white ppt. with Ba^{2+}(aq) or with Pb^{2+}(aq) (insoluble in excess dilute strong acid)
sulfite, SO_3^{2-} (aq)	SO_2 liberated with dilute acids; gives white ppt. with Ba^{2+}(aq) (soluble in excess dilute strong acid)

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
sulfur dioxide, SO_2	turns acidified aqueous potassium dichromate(VI) from orange to green

