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

CANDIDATE NAME	

CENTRE NUMBER						CANDIDATE NUMBER					

8263371454

CHEMISTRY **9701/33**

Paper 31 Advanced Practical Skills **May/June 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 15 and 16.

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 **14** printed pages and **2** blank pages.

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UNIVERSITY of CAMBRIDGE International Examinations

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1 You are required to determine the molar enthalpy change of solution for ammonium chloride, **FA 1**.

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When an exothermic reaction takes place in a container such as a beaker, some of the evolved heat energy is absorbed by the beaker.

When an endothermic reaction takes place some of the required heat energy is supplied by the beaker.

The amount of heat energy evolved or supplied for a 1 °C change in temperature is known as the heat capacity of the beaker.

In preparation for your experiment to determine the molar enthalpy change of solution for **FA 1** you will first need to determine the approximate heat capacity of a 250 cm^3 beaker.

Before starting any practical work read through the instructions in (a) and draw up a table to record your results.

(a) Determining the approximate heat capacity of the 250 cm^3 beaker

For Examiner's Use

When samples of hot and cold water are mixed in the 250 cm^3 beaker, some heat is lost to the beaker in raising its temperature. To determine the approximate heat capacity of your 250 cm^3 beaker, you will determine the maximum temperature rise when a sample of hot water is added to cold water in the beaker.

- Use a 50 cm^3 measuring cylinder to transfer 50 cm^3 of cold water into the 250 cm^3 beaker.
- Use the 50 cm^3 measuring cylinder to transfer 50 cm^3 of cold water into a 100 cm^3 beaker. Note the temperature of the water in this 100 cm^3 beaker and heat it **carefully and gently** until the temperature of the water in it has increased by 45–50 °C then stop heating, *e.g. if the water is at 20.0 °C you should warm it to 65–70 °C*.

- Stir the cold water in the 250 cm^3 beaker with the thermometer.
- Record the temperature of the cold water (this is the temperature at t = 0 min).
- Record the temperature each minute for 3 minutes.

- After you have taken the reading at t = 3 min, use the thermometer to stir the hot water in the 100 cm^3 beaker.
- At t = 4 min, measure the temperature of the hot water and record this value in the box below.
- **Immediately** add the hot water from the 100 cm^3 beaker to the cold water in the 250 cm^3 beaker. Stir with the thermometer but do **not** record the temperature.
- Continue to stir the water throughout the experiment.
- Record the temperature at t = 5 min, and then every **½ minute** until t = 8 min.
- Empty and rinse the 250 cm^3 beaker. Dry it using a paper towel.
- Record all measurements of time and temperature obtained.

The temperature, $\boldsymbol{T_1}$, of the hot water at t = 4 min is ………… °C.

Table of results

[3]

For Examiner's Use

(b) Graph plotting

1. Plot a graph of the temperature of the water in the 250 cm^3 beaker (y-axis) against time (x-axis) on the grid below.
   Do **not** plot the temperature, $\boldsymbol{T_1}$, of the hot water at t = 4 min.
2. Draw two straight lines of best fit; one through the points up to t = 3 min; the second through the points from t = 5 min to t = 8 min. Extrapolate both lines to t = 4 min.
3. From the extrapolated lines read the minimum and the maximum temperatures at t = 4 min. Record these values in the spaces provided below.
4. Determine the values for the two temperature changes at t = 4 min.

Minimum temperature, $\boldsymbol{T_2}$, at t = 4 min is .... °C.

Maximum temperature, $\boldsymbol{T_3}$, at t = 4 min is .... °C.

Temperature rise for 50 cm^3 of cold water in the 250 cm^3 beaker, ($\boldsymbol{T_3} - \boldsymbol{T_2}$) is .... °C.

Temperature fall for 50 cm^3 of hot water from the 100 cm^3 beaker, ($\boldsymbol{T_1} - \boldsymbol{T_3}$) is .... °C.

[4]

(c) Calculations

For Examiner's Use

Working should be shown in all calculations.

[**4.2 J** are absorbed or released when the temperature of 1.0 cm^3 of **water** changes by 1.0 °C.]

(i) Calculate the heat energy gained by the 50 cm^3 of cold water in the 250 cm^3 beaker.

The heat energy gained by the cold water = ………… J.

(ii) Calculate the heat energy lost by the 50 cm^3 of hot water from the 100 cm^3 beaker.

The heat energy lost by the hot water = ………… J.

(iii) The difference between the values calculated in **(i)** and **(ii)** is an approximate value for the total heat energy absorbed by the 250 cm^3 beaker during the experiment. The heat capacity of the beaker is the amount of heat energy absorbed for a 1 °C change in temperature.

$$\text{approximate heat capacity of the } 250\,\text{cm}^3 \text{ beaker} = \frac{(\textbf{heat energy lost}) - (\textbf{heat energy gained})}{(\boldsymbol{T_3} - \boldsymbol{T_2})}\ \text{J}\,°\text{C}^{-1}$$

Use your answers to **(i)** and **(ii)** and the temperature rise from **(b)** to calculate the approximate heat capacity of the 250 cm^3 beaker.

The approximate heat capacity of the 250 cm^3 beaker = ………… J °C^{-1}.

[1]

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(d) Determining the enthalpy change of solution for ammonium chloride

Follow the instructions below to find the temperature change when a known mass of solid ammonium chloride dissolves in water.

You are provided with two samples of ammonium chloride. You should use the sample labelled $\mathbf{NH_4C\mathit{l}}$ in **experiment 1** and the sample labelled **FA 1** in **experiment 2**.

Experiment 1

- Enter all results in the table below.
- Weigh the stoppered tube containing ammonium chloride, which is labelled $\mathbf{NH_4C\mathit{l}}$.
- Use the 50 cm^3 measuring cylinder to transfer 100 cm^3 of cold water into the rinsed and dried 250 cm^3 beaker used in **(a)**.
- Stir the water in the beaker with the thermometer and record the temperature.
- Add the solid from the weighed tube to the water.
- Stir the mixture constantly with the thermometer.
- Record the minimum temperature obtained in the solution.
- Reweigh the tube labelled $\mathbf{NH_4C\mathit{l}}$, its stopper and any residual ammonium chloride.
- Empty and rinse the beaker and dry it using a paper towel.

Experiment 2

- Enter all results in the table below.
- Weigh a clean, dry, boiling-tube.
- Weigh between 9.8 g and 10.2 g of **FA 1**, ammonium chloride, into the boiling-tube.
- Repeat the procedure in **experiment 1** and record the minimum temperature obtained when this mass of **FA 1** dissolves in 100 cm^3 of water.
- Reweigh the boiling-tube and any residual ammonium chloride.

Results

	experiment 1	experiment 2
mass of tube + ammonium chloride / g		
mass of empty tube / g		
mass of tube + residual ammonium chloride / g		
mass of ammonium chloride / g		
initial temperature of water / °C		
minimum temperature obtained / °C		
temperature fall, $\Delta \boldsymbol{T}$ / ^{o}C		

[6]

(e) Calculations

For Examiner's Use

Working should be shown in all calculations.

(i) Use the temperature fall from **(d)**, **experiment 1**, to calculate the change in heat energy of the solution.
[**4.3 J** are absorbed or released when the temperature of 1.0 cm^3 of **solution** changes by 1.0 °C.]

The change in heat energy of the solution = ... J.

(ii) To calculate the total change in heat energy as ammonium chloride dissolves in water, the change in heat energy of the 250 cm^3 beaker has to be added to the change in heat energy of the solution.
Explain why these two changes in heat energy have to be added together.

....

....

....

(iii) Use your answer in **(i)** above and the approximate heat capacity of the 250 cm^3 beaker calculated in **(c)(iii)** to calculate the combined change in heat energy of the beaker and solution.

The combined change in heat energy of the beaker and solution = ... J.

(iv) Calculate how many moles of **FA 1**, NH_4Cl, were used in **(d)**, **experiment 1**.
[A_r; C*l*, 35.5; H, 1.0; N, 14.0]

………… mol of **FA 1** were used in **experiment 1**.

i	
ii	
iii	
iv	

(v) Calculate the enthalpy change when 1 mol of **FA 1** dissolves in an excess of water. This is the molar enthalpy change of solution, $\Delta \boldsymbol{H}_{solution}$ (NH_4Cl).
Make certain that your answer is given in $kJ\,mol^{-1}$ and has the appropriate sign.

For Examiner's Use

$\Delta \boldsymbol{H}_{solution}$ (NH_4Cl) = ... $kJ\,mol^{-1}$.
sign *calculated value*

(vi) Explain the significance of the sign you have given in **(v)** and how it is related to your experimental results.

....

....

....
[8]

v	
vi	
vii	
viii	

For Examiner's Use

(f) Evaluation

A data book value for the molar enthalpy change of solution, $\Delta \boldsymbol{H}_{solution}$ (NH_4Cl), is +15.2 kJ mol^{-1}.

The value you have obtained may be significantly different from this value.

Calculate the difference between your value of $\Delta \boldsymbol{H}_{solution}$ (NH_4Cl) and the data book value. Record this difference below. Express this difference as a percentage of the data book value.

difference = ……………….. kJ mol^{-1}

percentage difference = ……………….. %

[1]

(g) Sources of error

Describe **one** major source of error in this experiment. Suggest an improvement which would significantly increase the accuracy of the experiment. Explain why your suggestion would produce a more accurate value.

description of major source of error

....

....

suggested improvement

....

....

explanation of why suggestion would increase experimental accuracy

....

....

[2]

[Total: 25]

For Examiner's Use

2 **(a)** You are provided with three solutions, **FA 2**, **FA 3** and **FA 4**. The only anions that may be present in these solutions are sulfate and carbonate. One or more of the solutions may contain both anions.

Identification of the anions in FA 2, FA 3 and FA 4.

Most metal carbonates are insoluble, most metal sulfates are soluble and all metal nitrates are soluble in water.

(i) Use this information and the Qualitative Analysis Notes on page 16 to select
- **reagent 1**, to identify any carbonate ion present,
- **reagent 2**, to identify any sulfate ion present.

reagent 1 .... **reagent 2** .... [1]

(ii) Explain the order in which you will add your chosen reagents to determine the anion or anions present in each of **FA 2**, **FA 3** and **FA 4**.

....

....

....[1]

(iii) Use the reagents selected in **(a)(i)** to test each of the solutions **FA 2**, **FA 3** and **FA 4**. Record your observations in the table below.

reagent	observations		
	FA 2	**FA 3**	**FA 4**

[2]

(iv) From your observations, identify the anion or anions present in each of the solutions.

solution	carbonate	sulfate	evidence
FA 2			
FA 3			
FA 4			

[2]

(b) You are provided with four solutions, **FA 5**, **FA 6**, **FA 7** and **FA 8**. Perform the test-tube experiments described below and record your observations in the table. Where gases are released they should be identified by a test, **described in the appropriate place in your observations.**

tests		observations			
		FA 5	**FA 6**	**FA 7**	**FA 8**
(i)	To about 1 cm depth of solution in a test-tube, add 10 drops of aqueous sodium hydroxide. Shake the mixture,				
	then add a further 2 cm depth of aqueous sodium hydroxide. Shake the mixture again,				
	then leave the tube to stand for 2–3 minutes.				
(ii)	If no precipitate has formed in test **(i)** above; transfer the solution to a **boiling-tube** and warm gently with a Bunsen burner. **Care: heating aqueous sodium hydroxide in a tube may cause the solution to be ejected from the tube**.				
(iii)	To about 1 cm depth of solution in a test-tube, add 10 drops of aqueous ammonia. Shake the mixture,				
	then add a further 2 cm depth of aqueous ammonia. Shake the mixture again,				
	then leave the tube to stand for 2–3 minutes.				

[7]

For Examiner's Use

i	ii	iii	iv	v	vi	vii

For Examiner's Use

(c) From your observations in **(b)**, identify the cation present in each of the following solutions.

solution	cation	evidence
FA 5		
FA 6		
FA 8		

[2]

[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 Al foil
nitrite, NO_2^- (aq)	NH_3 liberated on heating with OH^-(aq) and Al foil; NO liberated by dilute acids (colourless NO $\rightarrow$ (pale) brown NO_2 in air)
sulfate, SO_4^{2-} (aq)	gives white ppt. with Ba^{2+}(aq) (insoluble in excess dilute strong acid) or gives white ppt. with Pb^{2+}(aq)
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) (aq) from orange to green