



Teachers' Version

CATHOLIC JUNIOR COLLEGE

H2 CHEMISTRY 9729

2020 JC2 PRACTICAL HANDBOOK – Book 3

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Name: (2T)

Date:

Practical Worksheet 12: Determining Enthalpy Change by Calorimetry & Acid-Base Titration Pre-Lab Activity

Prior to your practical session for **Experiment 12**, you are required to do the following:

1. Read through the instructions and questions in Practical Worksheet 12.
2. Revise your lab techniques by viewing the videos below and answering the accompanying questions.

Please submit this pre-lab worksheet when you report for your practical session.

In part A2 of Practical Expt 12, you will be performing an acid-base titration.

Watch the following videos to refresh your memory on how to set up and perform a titration.

Part I Use of burette

Video Title: Titration Step 2 – Preparing the burette

<http://tinyurl.com/WS-1-Burette>

Duration: 1:26 min

Note: at 0:13, the burette should be filled below eye-level.



1. How do you get rid of air bubbles in the tip of the burette?

Open the tap and let the solution run until the air bubble is flushed out.

2. When taking the burette reading, read from the bottom of the meniscus for colourless solution and top of the meniscus for coloured solution.

3. How many decimal places do you have to record your burette readings to? 2

Part II Use of pipette

Video Title: Titration Step 3 – Using the pipette

<http://tinyurl.com/WS-1-Pipette>

Duration: 1:17 min

Note: at 0:17, the pipette should be held at the blue marking when inserting the pipette filler, and not at the bulb (for safety reasons)



4. After initial washing with tap water and rinsing with deionised water, which apparatus will require further rinsing with the solution it is to be filled with?

Circle the apparatus (can be more than one):

standard (volumetric) flask, burette, pipette, conical flask

Part III **Titration Experimental Techniques**
Video Title: Titration Step 4 – Performing the Titration
<http://tinyurl.com/WS-1-Screened-MO>
Duration: 2:40 min



5. There is an error in the volume of indicator used in the video (0:06 to 0:11 min). How many drops of indicator should you use instead? 1-2 drops

6. What is the purpose of the white tile?

To make it easier to see the colour change in the solution when the end-point is reached

7. If you are a right-hander, you should control the burette tap with your left hand and swirl the conical flask with your right hand. Vice-versa for left-hander.

8. In the video (1:00 to 1:15 min), the instructor rinsed the conical flask with deionised water when some titrant solution was splashed onto the inside of the conical flask. Will the titre values be affected? Give a reason for your answer.

No. The number of moles of titrant added is unaffected.

9. How do you know you are approaching the end-point of the titration?

The colour observed upon adding the titrant takes a longer time to disappear upon swirling

10. What would you need to do with the titrant when approaching the end-point?

add the titrant drop-wise

11. How many times do you repeat the titration?

At least two consistent titre values are obtained (i.e. within 0.10 cm³ of each other)

12. In Practical Expt 12, screened methyl orange indicator is used for the titration. Describe the colour change expected at the end-point. [Note that in this experiment, the excess unreacted base is in the conical flask while the acid (titrant) is added from the burette.]

Colour change at end-point: green to grey

13. In Practical Expt 12, the solution from **A1** is diluted (by making a standard solution in the volumetric flask) before titration in **A2**.

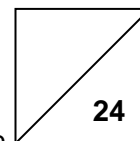
Why is this necessary?

To adjust the concentration of analyte such that at the end-point, the volume of titrant needed is about the same as the volume of analyte pipetted for titration (i.e. around 25 cm³)

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Date:

Catholic Junior College
H2 Chemistry (9729)

**Experiment 12: Determining Enthalpy Change by Calorimetry & Acid-Base Titration**

In this experiment, you are to determine the enthalpy change of solution for

- (i) anhydrous sodium carbonate (Na_2CO_3),
(ii) hydrated sodium carbonate ($\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$), and hence determine the enthalpy change for the conversion of anhydrous form into the hydrated form.

You will determine the quantities of sodium carbonate used by weighing and also by titrating with standard hydrochloric acid.

ANHYDROUS SODIUM CARBONATE

A1 Using an electronic balance, weigh accurately about 5 g of **anhydrous sodium carbonate** (Na_2CO_3). Record the relevant mass values in (i) of **Table 1**

[1] mass readings to 2 or 3 d.p. and mass weighed out in (i) within 4.95-5.04 g

Table 1

(i)	Mass of empty weighing bottle / g	3.900
	Mass of weighing bottle + anhydrous sodium carbonate / g	8.920
(ii)	Mass of emptied weighing bottle + residual anhydrous sodium carbonate / g	3.920
	Mass of anhydrous sodium carbonate used / g	5.000

Pour about 100 cm³ of deionised water into a beaker provided. Now, using a 25.0 cm³ pipette, transfer 50 cm³ of deionised water to the plastic (styrofoam) cup provided and note the temperature using the thermometer provided. Record the temperature in **Table 2** below.

Empty your sample of anhydrous sodium carbonate into the 50 cm³ of deionised water, stirring the solution as you do so. Record the highest temperature of the solution using the same thermometer.

Table 2

Highest temperature of solution / °C	35.4
Initial temperature of 50 cm ³ of deionised water / °C	29.8
Temperature change / °C	5.6

Stir the sodium carbonate solution thoroughly and then pipette a 25.0 cm³ sample into a 250 cm³ standard or volumetric flask provided. Make the solution up to the mark with deionised water. Shake this solution thoroughly and label it **FA1**.

Reweight the emptied weighing bottle and record the value in line (ii) of **Table 1** and then find the mass of anhydrous sodium carbonate added.

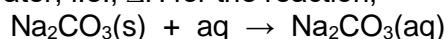
[1] temperature recorded to 1 d.p.
[2] accuracy: compare with tr's $\Delta T / m$
(2) if difference ≤ 0.30 °C g⁻¹
(1) if difference between 0.30 and 0.60 °C g⁻¹

- (a) If 210 J are required to raise the temperature of the 50 cm³ of sodium carbonate solution by 1 °C, calculate the heat evolved which occurred in dissolving your sample of anhydrous sodium carbonate.

$$\text{Heat evolved} = (210 \times 5.6) \text{ J} \quad [1] \quad 210 \times \Delta T$$

$$= 1176 \text{ J}$$

- (b) Hence, calculate the enthalpy change when 1 mol of anhydrous sodium carbonate ($M_r = 106.0$) is dissolved in water, i.e., ΔH for the reaction,



$$\text{Mol of Na}_2\text{CO}_3 = \left(\frac{5.000}{106.0} \right) = 0.0472 \text{ mol}$$

$$\Delta H = - \left(\frac{1176}{0.0472} \right) \text{ J mol}^{-1} \quad [1] \quad \text{ans in (a) / (correctly calculated) mol of Na}_2\text{CO}_3, \text{ with sign}$$

$$= - 24.9 \text{ kJ mol}^{-1}$$

- A2** Pipette 25.0 cm³ of **FA1** into a conical flask. Add one or two drops of the indicator, screened methyl orange, provided. Then titrate it with 0.200 mol dm⁻³ hydrochloric acid, **FA2**, with continuous swirling as you do so. The end-point is reached when the change in colour from green to grey, is observed in the conical flask. Repeat the titration as many times as you think necessary to achieve accurate results. Record the results of your titrations in the table below.

Titration of FA1 with FA2

Titration number	1	2	3
Final burette reading / cm ³	23.70	47.30	
Initial burette reading / cm ³	0.00	23.70	
Volume of FA2 used / cm ³	23.70	23.60	
Chosen titre values (indicate with a '✓')	✓	✓	

Summary

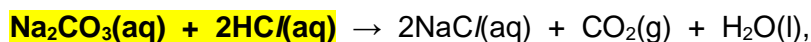
25.0 cm³ of **FA1** required 23.65 cm³ of **FA2** for reaction.

Show clearly how you obtained this value of the volume of **FA2** for reaction.

$$\text{Volume of FA2} = \left(\frac{23.70 + 23.60}{2} \right) = 23.65 \text{ cm}^3$$

- [1] readings recorded to 2 d.p., average titre vol calculated to 2 d.p.
 [1] at least 2 consistent titres (within $\pm 0.10 \text{ cm}^3$)
 [2] accuracy: compare with teacher's $\frac{\text{average titre vol}}{m_1}$
 (2) if difference $\leq 0.05 \text{ cm}^3 \text{ g}^{-1}$
 (1) if difference between 0.05 and $0.10 \text{ cm}^3 \text{ g}^{-1}$

- (c) By using these titration results, and given that the equation for the neutralization is



calculate the concentration, in mol dm^{-3} , of **FA1**.

$$\text{Mol of HCl} = \left(\frac{23.65}{1000} \times 0.200 \right) = 0.00473 \text{ mol} \quad [1] \quad \frac{\text{average titre}}{1000} \times 0.200$$

$$\text{Mol of Na}_2\text{CO}_3 \text{ in FA1} = \left(\frac{1}{2} \times 0.00473 \right) = 0.00237 \text{ mol}$$

$$\begin{aligned} \text{Concentration of Na}_2\text{CO}_3 \text{ in FA1} &= \left(0.00237 \times \frac{1000}{25.0} \right) \quad [1] \quad \frac{1}{2} \times \text{mol of HCl} \times \frac{1000}{25.0} \\ &= 0.0946 \text{ mol dm}^{-3} \end{aligned}$$

- (d) By using your answer from (c), calculate the concentration, in mol dm^{-3} , of your sodium carbonate solution in **A1**.

$$\text{Concentration of Na}_2\text{CO}_3 \text{ in A1} = \left(0.0946 \times \frac{250}{25.0} \right) = 0.946 \text{ mol dm}^{-3} \quad [1]$$

$$\text{(Note: Solution is diluted 10 times or } \frac{250}{25.0} \text{)}$$

- (e) By using your answer from (d), calculate the mass of anhydrous sodium carbonate used in **A1**.

$$\text{Mol of Na}_2\text{CO}_3 \text{ in A1} = \left(0.946 \times \frac{50.0}{1000} \right) = 0.0473 \text{ mol}$$

$$\text{Mass of Na}_2\text{CO}_3 \text{ used in A1} = (0.0473 \times 106.0) = 5.01 \text{ g} \quad [1]$$

(Note: Anhydrous Na_2CO_3 is contained in 50.0 cm^3 of solution in A1)

- (f) Does your answer to (e) agree with the weighings recorded in **Table 1**? What does this tell you about the sample of anhydrous sodium carbonate used?

Yes.

The sample is pure or has a high percentage of purity. [1]

HYDRATED SODIUM CARBONATE

B1 A similar procedure as in **A1** was carried out by a student but **hydrated sodium carbonate ($\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$)** was used instead. A stoppered vial containing **hydrated sodium carbonate (Na_2CO_3)** was weighed using an electronic balance and its value was recorded in line (i) of **Table 3** below.

Table 3

(i)	Mass of stoppered vial + hydrated sodium carbonate / g	17.864
(ii)	Mass of emptied stoppered vial + residual hydrated sodium carbonate / g	3.862
	Mass of hydrated sodium carbonate used / g	14.002

About 100 cm³ of deionised water was poured into a beaker provided. Using a 25.0 cm³ pipette, 50 cm³ of deionised water was transferred to the plastic (styrofoam) cup and its temperature was noted using the thermometer provided. The temperature was recorded in **Table 4** below.

The student emptied his sample of hydrated sodium carbonate into the 50 cm³ of deionised water, stirring the solution as he did so. The lowest temperature of the solution was then recorded using the same thermometer.

Table 4

Initial temperature of 50 cm ³ of deionised water / °C	30.2
Lowest temperature of solution / °C	14.4
Temperature change / °C	15.8

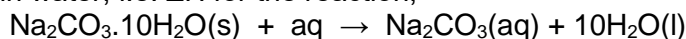
The sodium carbonate solution was thoroughly stirred and then 25.0 cm³ of the sample was pipetted into a 250 cm³ standard or volumetric flask provided. The solution was then made up to the mark with deionised water. This solution was shaken thoroughly and labelled **FA3**.

The emptied, stoppered vial was then reweighed and its value was recorded in line (ii) of Table 3 and the mass of hydrated sodium carbonate added was determined.

- (a) If 210 J are required to raise the temperature of the 50 cm³ of sodium carbonate solution by 1 °C, calculate the heat absorbed which occurred in dissolving the sample of hydrated sodium carbonate.

$$\begin{aligned} \text{Heat absorbed} &= (210 \times 15.8) \text{ J} & [1] & 210 \times \Delta T \\ &= 3318 \text{ J} \end{aligned}$$

- (b) Hence, calculate the enthalpy change when 1 mol of hydrated sodium carbonate ($M_r = 286.0$) is dissolved in water, i.e. ΔH for the reaction,



$$\text{Mol of Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O} = \left(\frac{14.002}{286.0} \right) = 0.0490 \text{ mol}$$

$$\Delta H = + \left(\frac{3318}{0.0490} \right) \text{ J mol}^{-1}$$

$$= + 67.8 \text{ kJ mol}^{-1}$$

[1] ans in (a) / (correctly calculated) mol of $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$, with sign

- B2** The same procedure as in **A2** was carried out by a student but **hydrated sodium carbonate ($\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$)** was used instead. 25.0 cm^3 of **FA3** was pipetted into a conical flask. One or two drops of the indicator, screened methyl orange, provided was added and then titrated with $0.200 \text{ mol dm}^{-3}$ hydrochloric acid, **FA2**. The results of titration were tabulated as below.

Titration of FA3 with FA2

Titration number	1	2	3
Final burette reading / cm^3	27.85	27.90	
Initial burette reading / cm^3	0.00	0.00	
Volume of FA2 used / cm^3	27.85	27.90	
Chosen titre values (indicate with a '✓')	✓	✓	

Summary

25.0 cm^3 of **FA3** required 27.88 cm^3 of **FA2** for reaction.

Show clearly how you obtained this value of the volume of **FA2** for reaction.

$$\text{Volume of FA2} = \left(\frac{27.85 + 27.90}{2} \right) = 27.88 \text{ cm}^3$$

[1] choice of titre values (ticked) & average titre vol calculated to 2 d.p.

- (c) By using these titration results, calculate the concentration, in mol dm^{-3} , of **FA3**.

$$\text{Mol of HC} = \left(\frac{27.88}{1000} \times 0.200 \right) = 0.00558 \text{ mol}$$

$$[1] \frac{\text{average titre}}{1000} \times 0.200$$

$$\text{Mol of Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O in FA3} = \left(\frac{1}{2} \times 0.00558 \right) = 0.00279 \text{ mol}$$

$$\begin{aligned} \text{Concentration of Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O in FA3} &= \left(0.00279 \times \frac{1000}{25.0} \right) \\ &= 0.112 \text{ mol dm}^{-3} \end{aligned} \quad [1] \quad \frac{1}{2} \times \text{mol of HCl} \times \frac{1000}{25.0}$$

- (d) By using your answer from (c), calculate the concentration, in mol dm^{-3} , of the sodium carbonate solution in **B1**.

$$\text{Concentration of Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O in B1} = \left(0.112 \times \frac{250}{25.0}\right) = 1.12 \text{ mol dm}^{-3} \text{ [1]}$$

$$\text{(Note: Solution is diluted 10 times or } \frac{250}{25.0} \text{)}$$

- (e) By using your answer from (d), calculate the mass of hydrated sodium carbonate used in **B1**.

$$\text{Mol of Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O in B1} = \left(1.12 \times \frac{50.0}{1000}\right) = 0.0558 \text{ mol}$$

$$\text{Mass of Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O used in B1} = (0.0559 \times 286.0) = 15.9 \text{ g [1]}$$

(Note: Hydrated $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ is contained in 50.0 cm^3 of solution in B1)

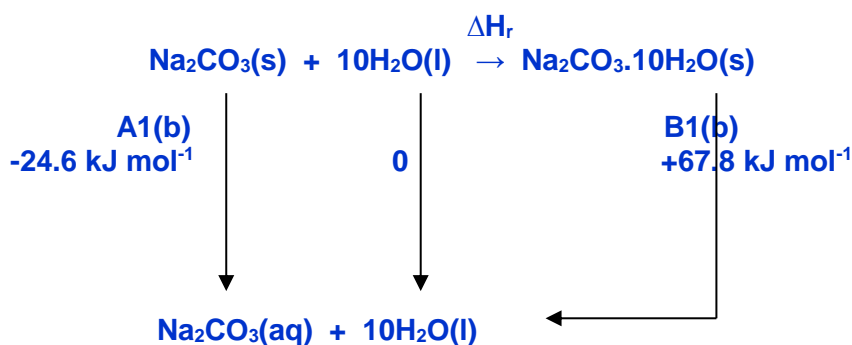
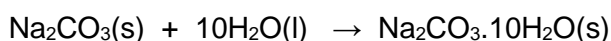
- (f) Does your answer to (e) agree with the weighings recorded in **Table 3**? How can you account for any difference?

No.

Efflorescence or loss of water of crystallisation takes place.

Or, hydrated sodium carbonate is no longer $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ [1]

- (g) By using your results from **A1(b)** and **B1(b)**, calculate the change that occurs when 1 mol of anhydrous sodium carbonate is converted to hydrated sodium carbonate, i.e. ΔH_r for the reaction,



Hess' Law,

$$\Delta H_r + (+67.8) = -24.6 + 0$$

$$\Delta H_r = -92.4 \text{ kJ mol}^{-1} \text{ [1]}$$

Safety

- The hydrochloric acid used is **corrosive**. (Although the solution is dilute, you should mop up any spillages and rinse any solution off your skin or clothing with water).

[Total: 24]

Check list: As you do your practical work, check that you have done the following by putting a tick (✓) against each item stated.

1	Manipulative Skills	(✓)
Calorimetry	• Support plastic (Styrofoam) cup in a beaker.	
	• Transfer solid into plastic (Styrofoam) cup without spillage.	
	• Keep thermometer bulb immersed in the solution throughout (tilt cup if necessary).	
	• Stir with thermometer without spillage.	
	• Monitor temperature change while stirring.	
	• Read temperature to half of the smallest scale division on thermometer. ➤ For thermometer calibrated in 1 °C, read temperature to nearest 0.5 °C. ➤ For thermometer calibrated in 0.2 °C, read temperature to nearest 0.1 °C.	
Burette	• Fill up burette at eye level or lower to avoid spillage. (Safety)	
	• Do not climb on a stool when filling burette. (Safety)	
	• Clamp burette vertically.	
	• Ensure no air column in the burette (particularly, volume below tap).	
Pipette	• Use pipette filler to avoid accidental swallowing of chemicals, and handle pipette safely when fitting the pipette filler. (Safety)	
	• Adjust liquid level in the pipette with the tip of pipette above liquid surface.	
Titration	• Place conical flask on a white tile during titration.	
	• Titrate with continuous swirling.	
	• Add titrant dropwise near end-point.	
	• Stop titration at correct end-point (yellow to orange).	
	• Repeat titration until two consistent titres (within $\pm 0.10 \text{ cm}^3$) are obtained.	
2	Presentation of Data	
Readings	• Record temperature readings to 1 decimal place.	
	• Record pipette readings to 1 decimal place. (Precision)	
	• Record burette readings to 2 decimal places (to nearest 0.05 cm^3). (Precision)	
	• Put tick (✓) against chosen titres used to calculate average titre value.	
Calculations	• Show working in ALL calculations (including average titre value).	
	• Leave working answers to 3 s.f. (or a maximum of 4 s.f.).	
	• Leave final answers to 3 or 4 s.f. (unless otherwise stated).	
	• Include sign for ΔH value ('-' for exothermic reaction, '+' for endothermic reaction).	
	• Give appropriate units in all calculations.	

Name: (2T)

Date:

Practical Worksheet 13: Iodometric Titration

Pre-Lab Activity

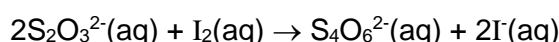
Prior to your practical session for **Experiment 13**, you are required to do the following:

1. Read through the instructions and questions in Practical Worksheet 13.
2. As you view the videos below, answer the accompanying questions.

Please complete and bring this worksheet before reporting for your practical session.

In Practical WS 13, you will be performing an iodometric titration.

The redox reaction involved in the reaction is:

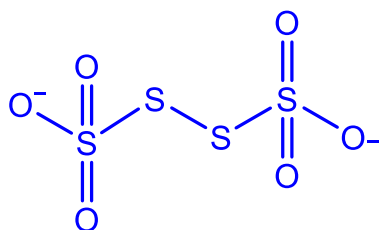


1. State the change in oxidation states of the reacting species.

O.S. of S increased from +2 in $\text{S}_2\text{O}_3^{2-}$ to +2.5 in $\text{S}_4\text{O}_6^{2-}$

O.S. of I decreased from 0 in I_2 to -1 in I^-

2. Given that there are three S-S bonds present in $\text{S}_4\text{O}_6^{2-}$, draw the structure of $\text{S}_4\text{O}_6^{2-}$.



3. Hence, suggest a possible reason why the average oxidation state of S in $\text{S}_4\text{O}_6^{2-}$ is not a whole number.

There are 2 S with oxidation states +5 and 2 S with oxidation states 0. The value of +2.5 is the average of these oxidation states.

4. In the experiment, a pipette is used to measure the volume of **FA 2** but a measuring cylinder is used for KI instead. Suggest a possible reason why this is so.

Cu^{2+} is the unknown and thus the volume measured needs to be precise so that an accurate value (i.e. mass) can be obtained. The KI used is in excess and thus accuracy of volume measured is not critical.

5. Why should starch indicator be added only when the solution becomes pale yellow?

When the concentration of iodine is high, a larger amount of iodine is trapped in the starch-iodine complex which results in the titre value being higher than expected.

Iodometric Titrations

Video Title: Iodine and sodium thiosulfate titrations
<http://tinyurl.com/WS10-iodometric>
Duration: 5:45 min
Stop the video at 5:45 min as the rest is not relevant



6. At 1:55min of the video, it was mentioned that there is a need to have a fixed amount of oxidising agent. What apparatus should be used to measure this fixed amount and why?

Being the unknown, and thus the limiting reagent, the volume measure must be precise.

Thus an apparatus of high precision such as pipette or burette should be used to ensure accurate results.

Iodometric Titrations

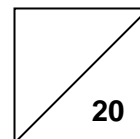
Video Title: Iodometric Titration
<http://tinyurl.com/WS10-colour>
Duration: 1 min 24 s



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Date:

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Experiment 13: Iodometric Titration

Copper forms compounds containing Cu^{2+} or Cu^+ ions. Those compounds containing Cu^{2+} ions tend to be relatively stable.

The addition of an excess of potassium iodide, KI, to a solution of Cu^{2+} ions produces iodine, I_2 , and a stable precipitate of CuI. For a titration to be accurate, it is necessary that all the Cu^{2+} ions are reduced to Cu^+ ions. The I_2 turns the solution brown.



I_2 has a relatively low solubility in water. However, the presence of an excess of I^- ions in the reaction mixture allows the soluble tri-iodide ion, I_3^- , to form as shown by equation 2.



The I_3^- ions formed may be titrated against a standard solution of $\text{Na}_2\text{S}_2\text{O}_3$ as shown in equation 3.



In this experiment, you will perform titrations to determine the percentage by mass of copper present in an alloy.

FA 2 is an aqueous solution containing 11.54 g dm^{-3} of the alloy in acid.

You are also provided with

FA 1, $0.100 \text{ mol dm}^{-3}$ sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$,
 0.5 mol dm^{-3} potassium iodide, KI,
aqueous sodium carbonate, Na_2CO_3 ,
aqueous ethanoic acid, $\text{CH}_3\text{CO}_2\text{H}$,
starch indicator.

The **FA 2** solution has been prepared using 11.54 g of the alloy containing copper and other metals, in 1 dm^3 of the solution. **FA 2** contains residual traces of acid.

The presence of acid in the titration mixture will affect the accuracy of the results. The procedure described is designed to reduce these errors.

In this experiment, you will determine the percentage by mass of copper in the alloy used to prepare **FA 2**. You will titrate **FA 2** against **FA 1**.

(a) (i) Titration of FA 2 against FA 1

1. Fill a burette with **FA 1**.
2. Use a pipette to transfer 25.0 cm³ of **FA 2** into a 250 cm³ conical flask.
3. Use a teat pipette (i.e., dropper) to add Na₂CO₃(aq) slowly, with shaking, to **FA 2** in the conical flask, until a slight permanent precipitate forms.
4. Use another teat pipette to add CH₃CO₂H(aq) slowly, with shaking, until this precipitate **just** dissolves.
5. Use a measuring cylinder to add about 20 cm³ of KI(aq) to this flask. A white precipitate forms in a brown solution.
6. Run **FA 1** from the burette into this flask. Near the end-point, when the brown solution becomes pale, add about 1 cm³ of starch indicator.
7. Continue adding **FA 1** slowly. The end-point is reached when the **solution** first becomes colourless. The white precipitate remains.
8. Record your titration results, to an appropriate level of precision, in the space provided.
9. Repeat points 2 to 8 until consistent results are obtained.
10. Record all of your burette readings and the volume of **FA 1** added in each titration in an appropriate form in the space below. Make certain any recorded results show the precision of your practical work.

Titration Results

	1	2
Final burette reading / cm ³	25.00	24.90
Initial burette reading / cm ³	0.00	0.00
Volume of FA 1 used / cm ³	25.00	24.90

✓

✓

[1] table with correct headers and units
[1] and correct recording of initial & final readings to 2 d.p. (nearest 0.05 cm³)
[1] at least two consistent titres (within ±0.10 cm³)

[3]

- (ii)** From your titrations, obtain a suitable volume of **FA 1**, $V_{\text{FA 1}}$, to be used in your calculations. Show clearly how you obtained this volume and place a tick (✓) under the readings used.

$$\text{average titre} = \frac{25.00 + 24.90}{2} = 24.95 \text{ cm}^3$$

[1] use consistent titres + ticks + 2 d.p.

[3] accuracy: compare titre value against teacher's titre
(3) if difference ≤ 0.3
(2) if difference between 0.3+ and 0.5
(1) if difference between 0.5+ and 0.8
(0) if difference > 0.8

$V_{\text{FA 1}} = \dots\dots\dots$
[4]

- (b) (i) Calculate how many moles of sodium thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$, were present in the average titre.

$$\begin{aligned}\text{mol of Na}_2\text{S}_2\text{O}_3 \text{ in average titre} &= 0.1 \times \frac{24.95}{1000} \\ &= 2.495 \times 10^{-3} \text{ mol}\end{aligned}$$

[1] $0.1 \times \frac{\text{average titre}}{1000}$

moles of $\text{Na}_2\text{S}_2\text{O}_3$ in average titre =
[1]

- (ii) Hence, calculate how many moles of Cu^{2+} were present in 25.0 cm^3 of FA 2.

Using equations 1, 2 and 3,



$$\begin{aligned}\text{mol of Cu}^{2+} \text{ in } 25 \text{ cm}^3 &= \text{mol of S}_2\text{O}_3^{2-} \\ &= 2.495 \times 10^{-3} \text{ mol}\end{aligned}$$

[1] ans to (b)(i)

moles of Cu^{2+} in 25.0 cm^3 =
[1]

- (iii) Calculate the mass of copper in the 11.54 g of the alloy used to prepare 1.00 dm^3 of FA 2 using your answer in (b)(ii).

Hence, determine the percentage by mass of copper in the alloy.

[Ar: Cu, 63.5]

mol of Cu^{2+} in 1 dm^3 FA 2

$$\begin{aligned}&= \frac{1000}{25.0} \times 2.495 \times 10^{-3} \\ &= 0.0998\end{aligned}$$

$$\text{mass of Cu in } 1 \text{ dm}^3 = 0.0998 \times 63.5 = 6.34 \text{ g}$$

[1] $\frac{1000}{25.0} \times 63.5 \times \text{mol of Cu}^{2+} \text{ in } 25.0 \text{ cm}^3$

mass of Cu in 11.54 g of the alloy =
[1]

$$\begin{aligned}\% \text{ by mass of Cu in the alloy} &= \frac{6.34}{11.54} \times 100\% \\ &= 54.9\% \text{ [1]}\end{aligned}$$

percentage by mass of copper in the alloy =
[1]

(c) A large excess of potassium iodide is used in each titration.

- (i) Calculate the ratio of the amount of iodide ions added in each titration to the minimum amount of iodide ions required to reduce the copper(II) ions used.

From equation 1,
 $2\text{I}^- \equiv \text{Cu}^{2+}$

$$\text{Min. amt of I}^- = 2 \times 2.495 \times 10^{-3} \\ = 4.99 \times 10^{-3}$$

$$\text{Amt of I}^- \text{ added} = \frac{20}{1000} \times 0.5 \\ = 0.01 \text{ [1 for both correct amt calculated]}$$

Ratio of amt of I^- added : min. amt of I^- required

$$0.01 : 4.99 \times 10^{-3}$$

$$2:1 \text{ [1]}$$

[1] clear statements & working in (b) and (c)(i)
 [1] answers given in (b) & (c)(i) to 3 or 4 s.f.; ratio in (c)(i) can be presented as whole no.
 [1] units included in answers in (b)

ratio =
 [5]

- (ii) Identify **two** different chemical processes that use iodide ions in this experiment.

The two different chemical processes are

redox reaction (equation 1) and

solvation/dissolution of I_2 (equation 2). [1] (*It is a solvent for I_2*)

[1]

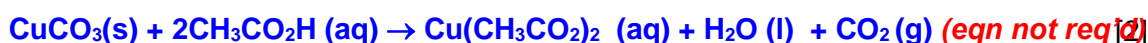
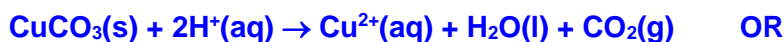
- (d) In (a)(i), $\text{Na}_2\text{CO}_3(\text{aq})$ was added to **FA 2** until a precipitate was formed, followed by $\text{CH}_3\text{CO}_2\text{H}(\text{aq})$ to dissolve this precipitate.

- (i) Explain the observations in terms of the chemistry involved.

When Na_2CO_3 is added, blue ppt of CuCO_3 was formed. [1]

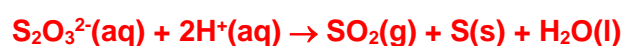


When $\text{CH}_3\text{CO}_2\text{H}$ was added, it reacts with the CuCO_3 ppt to form a soluble salt, causing the ppt to dissolve. [1]



- (ii) Explain why it was necessary to add $\text{Na}_2\text{CO}_3(\text{aq})$ to **FA 2**, and what effect failing to do it would have on the titre values.

The H^+ present reacts with $\text{S}_2\text{O}_3^{2-}$ to produce sulfur (ppt), therefore, more $\text{S}_2\text{O}_3^{2-}$ would be required to react with I_2 , causing the titre value to be higher. [1] (Recall this was a common question in Kinetics! – sulfur clock reaction)



[1]

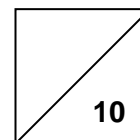
[Total: 20]

Check list: As you do your practical work, check that you have done the following by putting a tick (✓) against each item stated.

1	Manipulative Skills	(✓)
Burette	• Fill up burette at eye level or lower to avoid spillage. (Safety)	
	• Do not climb on a stool when filling burette. (Safety)	
	• Clamp burette vertically.	
	• Ensure no air column in the burette (particularly, volume below tap).	
Pipette	• Use pipette filler to avoid accidental swallowing of chemicals, and handle pipette safely when fitting the pipette filler. (Safety)	
	• Adjust liquid level in the pipette with the tip of pipette above liquid surface.	
Titration	• Place conical flask on a white tile during titration.	
	• Titrate with continuous swirling.	
	• Add titrant dropwise near end-point.	
	• Stop titration at correct end-point (blue-black to colourless).	
	• Repeat titration until two consistent titres (within $\pm 0.10 \text{ cm}^3$) are obtained.	
2	Presentation of Data	
Readings	• Draw table with appropriate headings and units.	
	• Record pipette readings to 1 decimal place. (Precision)	
	• Record burette readings to 2 decimal places (to nearest 0.05 cm^3). (Precision)	
	• Put tick (✓) against chosen titres used to calculate average titre value.	
Calculations	• Show working in ALL calculations (including average titre value).	
	• Leave working answers to 3 s.f. (or a maximum of 4 s.f.).	
	• Leave final answers to 3 or 4 s.f. (unless otherwise stated).	
	• Give appropriate units in all calculations.	

Name: (2T)

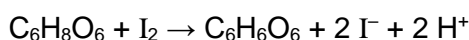
Date:

Catholic Junior College
H2 Chemistry (9729)

Experiment 13 (Planning): Iodometric Titration

Ascorbic acid is a naturally occurring organic compound commonly found in citrus fruits and Vitamin C supplements. It is also known as an 'anti-oxidant' or a reducing agent. Ascorbic acid is water-soluble. Direct iodometric titration can be used to determine the amount of ascorbic acid in samples of fruit juices and Vitamin C supplements.

Iodine rapidly oxidises ascorbic acid, $C_6H_8O_6$, to produce dehydroascorbic acid, $C_6H_6O_6$.



In the direct iodometric titration, the ascorbic acid is titrated against an iodine solution of known concentration from the burette, using starch as an indicator. Once all the ascorbic acid has been oxidised, the excess iodine is free to react with the starch indicator.

A student was given a Vitamin C supplement sample that the manufacturer claims to contain 0.25 g of ascorbic acid per tablet. She was tasked to plan an experiment to determine if the manufacturer's claim was true.

The following reagents were provided:

- FA 1** 0.00500 mol dm⁻³ I₂ solution
FA 2 starch indicator

- (i) The student was provided with a 250 cm³ volumetric flask to prepare an ascorbic acid solution using one Vitamin C tablet. The solution was labelled **FA 3**.

Assuming that the manufacturer's claim is true, calculate the concentration of ascorbic acid in **FA 3**. (5.68×10^{-3} mol dm⁻³) [1]

$$M_r \text{ of } C_6H_8O_6 = 12.0 \times 6 + 1.0 \times 8 + 16.0 \times 6 = 176.0$$

$$\text{No. of moles of ascorbic acid} = \frac{0.25}{176} = 1.42 \times 10^{-3} \text{ mol}$$

$$\text{Concentration of ascorbic acid in FA 3} = \frac{1.42 \times 10^{-3}}{\frac{250}{1000}} = \underline{5.68 \times 10^{-3} \text{ mol dm}^{-3}} \text{ [1]}$$

- (ii) Given that the average titre volume should be around 25.00 cm³, determine the minimum volume of **FA 3** that should be used in the experiment.

Hence, suggest a suitable pipette volume for titration. [3]

Assuming a titre volume of 25.00 cm³

$$\text{No. of moles of } I_2 \text{ in } 25.00 \text{ cm}^3 \text{ of FA 1} = \frac{25}{1000} \times 0.00500 = 1.25 \times 10^{-4} \text{ mol}$$

$$\text{No. of moles of ascorbic acid in FA 3 required for reaction} = 1.25 \times 10^{-4} \text{ mol [1]}$$

Volume of ascorbic acid in **FA 3** required for reaction

$$= \frac{1.25 \times 10^{-4}}{5.68 \times 10^{-3}} \times 1000 = 22.0 \text{ cm}^3 \text{ [1]}$$

$$\text{Volume of pipette required} = \underline{25.0 \text{ cm}^3} \text{ [1]}$$

(iii) Write a plan for the direct iodometric titration of ascorbic acid sample **FA 3**. You may use the reagents and apparatus normally found in a school or college laboratory. Your plan should include:

- details for the preparation of **FA 3** solution;
- the essential details of the titration procedure;
- the necessary calculation steps required to verify the manufacturer's claim

Objective of Plan:

- To determine the mass of ascorbic acid per tablet through iodometric titration

Technique/ Procedure + Choice of Apparatus:

- Prepare standard solution from solid sample: beaker, volumetric flask
- Titration: burette, conical flask, pipette
- Repeat experiment for consistent results: draw table

Type of measurement + Choice of Apparatus:

- Volume of ascorbic acid sample **FA 3**: 25 cm³ pipette
- volume of I₂ solution: 50 cm³ burette

Prepare standard solution from solid sample

1. Dissolve the Vitamin C tablet using deionised water in a 100 cm³ beaker.
2. Transfer the resultant solution and washings into a 250 cm³ volumetric flask and make up to mark with deionised water.
3. Shake well to obtain a homogenous solution and label it as **FA3**.

[1] preparation of standard solution technique with correct choice of apparatus

Titration

4. Pipette 25.0 cm³ of **FA3** solution using the 25.0 cm³ pipette and transfer it into a 250 cm³ conical flask.
5. Add 2–3 drops of **starch indicator** to the solution.
6. Titrate **FA3** solution with 0.00500 mol dm⁻³ I₂ solution from a 50 cm³ burette until the colour changes from **colourless** (due to no excess I₂ present in conical flask initially) to **blue-black** (due to one drop of excess I₂ in conical flask at end-point).
7. Repeat the titration until consistent results within ±0.10 cm³ are obtained.

[1] appropriate reagents in pipette/burette

[1] correct colour change @ endpt

[1] Repeat until consistent results

Table and summary statement titration readings

	1	2	
Final burette reading / cm ³			
Initial burette reading / cm ³			
Volume of I ₂ used / cm ³	V ₁	V ₂	

25.0 cm³ of the **standard ascorbic acid solution** required $\frac{V_1+V_2}{2}$ cm³ of **0.00500 mol dm⁻³ I₂ solution** for complete reaction.

[1] show how to obtain the average titre

Since C₆H₈O₆ ≡ I₂,

mass of ascorbic acid per tablet

= moles of C₆H₈O₆ in 25.0 cm³ FA3 × $\frac{250}{25}$ × M_r of C₆H₈O₆

= $\left(\frac{V_1+V_2}{2} \times 0.005 \right) \times \frac{250}{25} \times 176$

[1] show how to calculate the mass of ascorbic acid per tablet

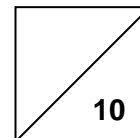
[6]

[Total: 10]

Name: (2T)

Date:

Catholic Junior College
H2 Chemistry (9729)



Experiment 14: Learning Experience on Buffers (Chemistry of Aq. Solns)

Objective: To investigate the effect on the pH of a buffer solution on adding different amounts of acid (HCl) and base (NaOH).

In this activity, you will prepare a buffer solution consisting of ethanoic acid, $\text{CH}_3\text{CO}_2\text{H}$, and sodium ethanoate, $\text{CH}_3\text{CO}_2\text{Na}$, and observe how the pH changes when different amounts of HCl and NaOH are added to separate samples of the buffer solution.

FA 1 is 0.5 mol dm^{-3} ethanoic acid, $\text{CH}_3\text{CO}_2\text{H}$

FA 2 is 0.5 mol dm^{-3} sodium ethanoate, $\text{CH}_3\text{CO}_2\text{Na}$

FA 3 is 1.5 mol dm^{-3} hydrochloric acid, HCl

FA 4 is 1.5 mol dm^{-3} sodium hydroxide, NaOH

[Caution: FA 3 and FA 4 are corrosive. Handle with care.]

Procedure (Part 1)

1. To prepare the buffer solution, mix 50 cm^3 of **FA 1** and 50 cm^3 of **FA 2** in a 250 cm^3 beaker labelled **buffer**. [Use different measuring cylinders.]

Place 100 cm^3 of water in another 250 cm^3 beaker.

Thinking Questions for Part 1

1. What type of buffer solution have you prepared?
acidic buffer
2. What is the reason for using equal volume of **FA 1** and **FA 2** in preparing the buffer?
buffer solution prepared has maximum buffer capacity (for most effective buffering action)

Procedure (Part 2)

2. Dip a piece of pH indicator paper into each beaker from step 1 and estimate the initial pH of each solution. Record your results in the table on the next page.
3. Use the test pipette (i.e., plastic dropper) to add 2 cm^3 of **FA 3** to each beaker. Mix the solution thoroughly with a glass rod and then estimate the pH of the resultant solution using the pH indicator (universal indicator) paper. Record your results in the table on the next page.
4. Repeat step 3 by adding 3 cm^3 , 5 cm^3 and 10 cm^3 of **FA 3** respectively, recording your results in the table on the next page.
5. Wash the two beakers. Prepare another set of buffer solution and water as in step 1.
6. Repeat steps 3 and 4 using **FA 4** in place of **FA 3**.

Results:

total vol of FA 3 / FA 4 added / cm ³	pH of resultant solution			
	with FA 3		with FA 4	
	buffer	water	buffer	water
0	4	5	4	5
2	3 or 4	2	4	14
2+3 = 5	3	1	4 or 5	14
5+5 = 10	3	1	5	14
10+10 = 20	1 or 2	1	10 to 12	14

Thinking Questions for Part 2

3. How did the pH of each solution change upon adding 2 cm³ of **FA 3** / **FA 4**?

buffer solution shows very little change in pH (0.5 units with FA 3 and hardly any change with FA4) whereas water shows a larger change in pH (3 units / 9 units respectively).

4. In each solution, what has happened to the H⁺/OH⁻ ions added to account for the pH change observed in Q3?

In buffer solution, H⁺/OH⁻ added are removed by large reservoir of CH₃CO₂⁻ ions/ CH₃CO₂H.

H⁺ + CH₃CO₂⁻ → CH₃CO₂H

OH⁻ + CH₃CO₂H → CH₃CO₂⁻ + H₂O

H⁺/OH⁻ added remain in water, thereby making the solution acidic (pH 2) / alkaline (pH 14).

5. What can you deduce about the buffering action in maintaining pH as different amounts of acid/base are added?

pH maintained only when small amounts of acid/base added. Buffering action lost when large amounts of acid/base added.

6. Instead of using pH indicator paper, what could you use to obtain more accurate pH values?

pH meter

7. Other than replacing the pH indicator paper, how would you modify the procedure to improve the overall accuracy of the experiment?

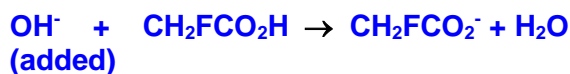
• pipette/burette to measure vol of FA 1 & FA 2;

• burette to measure vol of FA 3 and FA 4;

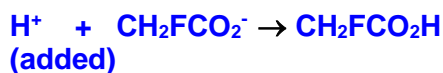
magnetic stirrer to keep solution well mixed.

Practice Questions

- 1 (a) By considering a solution containing both fluoroethanoic acid ($\text{CH}_2\text{FCO}_2\text{H}$) and sodium fluoroethanoate ($\text{CH}_2\text{FCO}_2\text{Na}$), explain how a buffer solution functions.



Small amount of OH^- ions are removed by the large concentration / reservoir of $\text{CH}_2\text{FCO}_2\text{H}$. Hence pH of solution remains almost unchanged.



Small amount of H^+ ions added are removed by the large concentration / reservoir of $\text{CH}_2\text{FCO}_2^-$ present. Hence pH of solution remains almost unchanged.

- (b) What would be the pH of three solutions whose concentrations of fluoroethanoic acid : sodium fluoroethanoate are respectively in the following molar ratios?

(K_a for fluoroethanoic acid is $2.51 \times 10^{-3} \text{ mol dm}^{-3}$.)

- (i) 1 : 1 (ii) 10 : 1 (iii) 1 : 10

$$(i) \quad \text{pH} = \text{p}K_a + \lg \frac{[\text{salt}]}{[\text{acid}]} = -\lg(2.51 \times 10^{-3}) + \lg\left(\frac{1}{1}\right) = \underline{2.60}$$

$$(ii) \quad \text{pH} = \text{p}K_a + \lg \frac{[\text{salt}]}{[\text{acid}]} = -\lg(2.51 \times 10^{-3}) + \lg\left(\frac{1}{10}\right) = \underline{1.60}$$

$$(iii) \quad \text{pH} = \text{p}K_a + \lg \frac{[\text{salt}]}{[\text{acid}]} = -\lg(2.51 \times 10^{-3}) + \lg\left(\frac{10}{1}\right) = \underline{3.60}$$

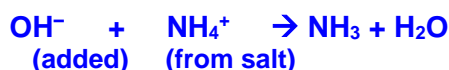
- 2 (a) (i) Explain what is meant by a *buffer solution*.

A buffer solution is a solution whose pH remains almost unchanged on dilution or when small amounts of acid or base are added to it.

- (ii) With the aid of two equations, explain how a solution of ammonium chloride and ammonia can control pH.



Small amount of H^+ ions are removed by the large concentration / reservoir of NH_3 . Hence pH of solution remains almost unchanged.



Small amount of OH^- ions added are removed by the large concentration / reservoir of NH_4^+ . Hence pH of solution remains almost unchanged.

- (b) The buffer solution is prepared as follows: 53.5 g of ammonium chloride, NH_4Cl , is dissolved in 400 cm^3 of 15.0 mol dm^{-3} ammonia and the mixture diluted to 1.00 dm^3 .

Calculate the concentration, in mol dm^{-3} , in the prepared buffer solution of

- (i) $[\text{NH}_4^+(\text{aq})]$ [A_r of N = 14.0, A_r of H = 1.0, A_r of Cl = 35.5]

$$M_r \text{ of } \text{NH}_4\text{Cl} = 14 + 4(1) + 35.5 = 53.5$$

$$\text{No. of mol of } \text{NH}_4^+ = \text{No. of mol of } \text{NH}_4\text{Cl} = \frac{53.5}{53.5} = 1.0$$

$$\therefore [\text{NH}_4^+] = \underline{1.0\text{ mol dm}^{-3}}$$

- (ii) $[\text{NH}_3(\text{aq})]$

$$\text{No. of mol of } \text{NH}_3 = 15.0 \times \frac{400}{1000} = 6.0$$

$$\therefore [\text{NH}_3] = \underline{6.0\text{ mol dm}^{-3}}$$

- (c) Use one of the following values to calculate the pH of the prepared buffer solution:

(ci)

$$\text{NH}_4^+(\text{aq}), K_a = 6.00 \times 10^{-10}\text{ mol dm}^{-3}$$

$$\text{NH}_3(\text{aq}), K_b = 1.67 \times 10^{-5}\text{ mol dm}^{-3};$$

- (i) Write a chemical equation, with an equilibrium sign, for the equilibrium you have chosen for the pH calculation.



- (ii) Write an expression, in terms of concentration, for the equilibrium constant, K_a or K_b for your chosen equilibrium.

$$K_b = \frac{[\text{NH}_4^+][\text{OH}^-]}{[\text{NH}_3]}$$

- (iii) Calculate the pH of the prepared buffer solution.

$$[\text{salt}] = [\text{NH}_4^+] = 1.0\text{ mol dm}^{-3}$$

$$[\text{base}] = [\text{NH}_3] = 6.0\text{ mol dm}^{-3}$$

$$\text{pOH} = \text{p}K_b + \lg \frac{[\text{salt}]}{[\text{base}]} = -\lg(1.67 \times 10^{-5}) + \lg\left(\frac{1.0}{6.0}\right) = 4.00$$

$$\therefore \text{pH} = 14 - \text{pOH} = 14 - 4.00 = \underline{10.0}$$