Planning Tutorial 1 : Volumetric Analysis (Suggested Answer)

Q2(a)

Pre-experiment calculations
Concentration of $Na_2S_2O_3$ in **FA 4** = 0.0400 mol dm^{-3}
Amount of $Na_2S_2O_3$ in 250 cm^3 of **FA 4** = (250/1000)(0.0400) = 0.0100 mol
M_r of of $Na_2S_2O_3.5H_2O$ = 248.2
Mass of $Na_2S_2O_3.5H_2O$ to use = (0.0100)(248.2) = 2.482 = 2.48 g **[1]**

Procedure to prepare 250 cm^3 of FA 4
1. Using an electronic balance, weigh accurately about 2.48 g of $Na_2S_2O_3.5H_2O$ solid in a clean and dry weighing bottle.
2. Transfer the $Na_2S_2O_3.5H_2O$ solid from the weighing bottle to a 100 cm^3 beaker quantitatively. Rinse the weighing bottle a few times with deionised water and transfer the washings to the 100 cm^3 beaker.
3. Using the wash bottle, add some deionised water to the beaker to dissolve the $Na_2S_2O_3.5H_2O$ solid. Using a glass rod, stir the mixture to ensure that all the solid dissolves, adding more deionised water if necessary.
4. Transfer the solution quantitatively into a 250 cm^3 volumetric flask. Rinse the beaker a few times with deionised water and transfer the washings into the volumetric flask.
5. Top up the volumetric flask to the 250 cm^3 mark with deionised water, using a dropping pipette to add the deionised water drop-wise when nearing the mark.
6. Stopper the volumetric flask and shake the solution thoroughly to ensure that it is homogeneous. Label this solution as **FA 4**.

[1] weighing + dissolving + quantitative transfer
[1] correct use of volumetric flask

Q2(b)

Pre-experiment calculations
Amount of $Na_2S_2O_3$ in 250 cm^3 of **FA 4** = (250/1000)(0.0400) = 0.0100 mol
Volume of 0.250 mol dm^{-3} $Na_2S_2O_3$ solution needed = 0.0100/0.250 = 0.0400 dm^3 = 40.00 cm^3 **[1]**

Procedure to prepare 250 cm^3 of FA 4
1. Fill a burette with 0.250 mol dm^{-3} $Na_2S_2O_3$ solution. Record the initial burette reading.
2. Run from the burette 40.00 cm^3 of the $Na_2S_2O_3$ solution into a 250 cm^3 graduated flask. Record the final burette reading.
3. Top up the graduated flask to the 250 cm^3 mark with deionised water, using a dropping pipette to add the deionised water drop-wise when nearing the mark.
4. Stopper the graduated flask and shake the solution thoroughly to ensure that it is homogeneous. Label this solution as **FA 4**.

Final burette reading / cm^3	40.00
Initial burette reading / cm^3	0.00
Volume of 0.250 mol dm^{-3} $Na_2S_2O_3$ solution used / cm^3	40.00

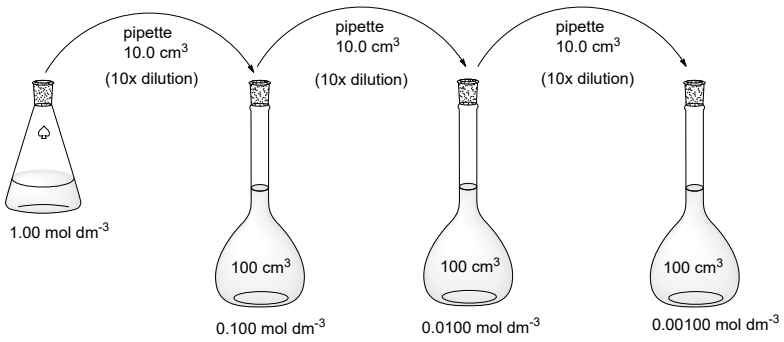
[1] correct use of burette + table
[1] correct use of volumetric flask

Q2(c)

Pre-experiment calculations
Dilution factor from 1.00 mol dm^{-3} to 0.100 mol dm^{-3} $Na_2S_2O_3$ = 10
Dilution factor from 0.100 mol dm^{-3} to 0.0100 mol dm^{-3} $Na_2S_2O_3$ = 10
Dilution factor from 0.0100 mol dm^{-3} to 0.00100 mol dm^{-3} $Na_2S_2O_3$ = 10

Hence, volume of 1.00 mol dm^{-3} solution to prepare 100 cm^3 of 0.100 mol dm^{-3} $Na_2S_2O_3$ = 10.0 cm^3
Volume of 0.100 mol dm^{-3} solution to prepare 100 cm^3 of 0.0100 mol dm^{-3} $Na_2S_2O_3$ = 10.0 cm^3
Volume of 0.0100 mol dm^{-3} solution to prepare 100 cm^3 of 0.00100 mol dm^{-3} $Na_2S_2O_3$ = 10.0 cm^3

[1] recognising 10x dilution / volume of 10 cm^3



Procedure
1. Pipette 10.0 cm^3 of 1.00 mol dm^{-3} sodium thiosulfate into a 100 cm^3 graduated flask.
2. Top up the graduated flask to the 100 cm^3 mark with deionised water, using a dropping pipette to add the deionised water drop-wise when nearing the mark.
3. Stopper the graduated flask and shake the solution thoroughly to ensure that it is homogeneous. The solution is 0.100 mol dm^{-3} sodium thiosulfate.
4. Repeat steps 1-3 using 10.0 cm^3 of 0.100 mol dm^{-3} sodium thiosulfate to prepare 0.0100 mol dm^{-3} sodium thiosulfate.
5. Repeat steps 1-3 using 10.0 cm^3 of 0.0100 mol dm^{-3} sodium thiosulfate to prepare 0.00100 mol dm^{-3} sodium thiosulfate.

[1] correct use of pipette + conc of sodium thiosulfate used
[1] correct use of volumetric flask

Q3(a)

General guiding questions

- Write an expression to determine the percentage purity by mass of $CaCO_3$ in the impure sample. What information do you need and how can this be obtained from the experiment?

  Percentage purity = $\frac{\text{mass of } CaCO_3}{\text{mass of sample used}}$ x 100%

  The amount and mass of $CaCO_3$ present in a known mass of impure sample needs to be determined via a back titration experiment.

- Why is the back titration method used instead of a direct titration method *(refer to Planning Experiments 1 - Volumetric Analysis worked example 1)*? What is the use of NaOH in this experiment?

  Since $CaCO_3$ is insoluble in water, a back titration is preferred as difficulties, e.g. accurate end-point determination, would arise if it were to be titrated with HCl directly. NaOH is required to react with the excess unreacted HCl in the back titration.

Field Code Changed

m g sample containing $CaCO_3$(s) + **FA 1** HCl(aq) **x cm³** 1.00 mol dm^{-3} (excess) → reaction → some HCl(aq) remained → H_2O → HCl(aq) 250 cm³ graduated flask → pipette → HCl(aq) 25.0 cm³ conical flask → titration → **FA 2** NaOH(aq) **y cm³** 0.100 mol dm^{-3}

$CaCO_3 + 2HCl \rightarrow CaCl_2 + CO_2 + H_2O$

$HCl + NaOH \rightarrow NaCl + H_2O$

Key Points	Remarks
Weighing of impure sample	• Method: Direct weighing or weighing by difference (weighing by difference method is used as sample is insoluble in water) • Recording: Mass measurements • Apparatus: Weighing bottle, analytical balance
Reaction of calcium carbonate	• Ensure complete reaction with HCl → HCl must be in excess • Measurement of HCl → volume needed and precision of apparatus • Reaction between $CaCO_3$ and HCl is complete when effervescence ceased. ○ Should the solid be added to **FA 1** or **FA 1** be added to the solid? (solid should be added to FA 1. Addition of FA 1 from the burette to the solid will result in greater loss of chemicals due to acid spray) • Loss of chemicals due to acid spray must be minimised (how?)
Preparation of sample solution and use of graduated flask	• Is there a need to prepare a solution using a graduated flask? If so, what is the capacity of the graduated flask to be used? (Graduated flask should be used – no need to prepare fresh sample for each titration. Capacity, usually 250 cm³, should be chosen to ensure sufficient solution for a few titrations to obtain consistent results) • Ensure quantitative transfer (how?) • Apparatus: 250 cm³ conical flask, burette (for HCl), filter funnel, glass rod, 250 cm³ graduated flask.
Titration	• Method: Back titration • Apparatus: Pipette (usually 25.0 cm³), burette, conical flask • Identity of solutions placed in conical flask or burette • Choice of indicator and end-point colour change • Consistent titre values (within 0.10 cm³) • Recording: Measurement of burette readings/titration results
Pre-calculations	*(You may find the diagram below useful for your pre-calculations)* • Determine the volume of reagents to be used ○ Set the titre value (i.e. 20.00 cm³ **FA 2** used) ○ Set the mass of impure sample used (i.e. 1.50 g) ○ Volume of **FA 1** by considering the amount of HCl that reacted with $CaCO_3$ and the amount of HCl present in excess in the graduated flask. • Are there any assumptions that need to be made? (The impure sample contains 80% by mass of $CaCO_3$ and any impurities present do not react with HCl and NaOH.)

- Percentage purity
  $= \frac{\text{mass of } CaCO_3}{\text{mass of sample used}} \times 100\%$
  The amount and mass of $CaCO_3$ present in a known mass of impure sample needs to be determined from the experiment.
- Since $CaCO_3$ is insoluble in water, a back titration is preferred as difficulties, e.g. accurate end-point determination, would arise if it were to be titrated with HC*l* directly.
- For the back titration, HC*l* (in excess) is required
  - to react with all the $CaCO_3$ present in the sample
  - to react with NaOH in the titration

- To find the total volume/ amount of HC*l* required, we need to consider the
  - amount of HC*l* needed to react with $CaCO_3$
    ⇒ state the mass of sample used and assume it contains 80% $CaCO_3$
  - amount of HC*l* (in 25.0 cm^3 solution) that reacted with NaOH in the titration
    ⇒ set the volume of titrant used (20.00 – 25.00 cm^3)
  - amount of HC*l* in 250 cm^3 solution (in graduated flask)

- Note: Any impurities present in the sample should not interfere with the experiment. In the case that the impurities are insoluble in water, a filtration would be required.

The reagent to be used in excess is the **1.00 mol dm^{-3} HC*l* (FA 1).**

A known excess of 1.00 mol dm^{-3} HC*l* (**FA 1**) can be added to the given sample of $CaCO_3$. After reaction with the $CaCO_3$, the unreacted HC*l* can be diluted and then titrated against 0.100 mol dm^{-3} NaOH (**FA 2**).

Pre-experimental calculations:

1. **Determine the amount of HC*l* required to react with $CaCO_3$**

Assumption: The 1.50 g sample contains 80% by mass of $CaCO_3$ and any impurities present do not react with HC*l* and NaOH.

Molar mass of $CaCO_3$ = 100.1 g mol^{-1}
Amount of $CaCO_3 = (\frac{1.50}{100.1})(0.8) = 0.01199$ mol

$CaCO_3(s) + 2HCl(aq) \longrightarrow CaCl_2(aq) + CO_2(g) + H_2O(l)$

Amount of HC*l* required to react with $CaCO_3$
= (2)(0.01199) = 0.02398 mol

2. **Determine the amount of HC*l* required to react with NaOH during titration**

Let the volume of NaOH used be 20.00 cm^3.
Amount of NaOH = (20.00/1000)(0.100) = 2.00 x 10^{-3} mol

$NaOH(aq) + HCl(aq) \longrightarrow NaCl(aq) + H_2O(l)$

Amount of HC*l* in 25.0 cm^3 of solution = 2.00 x 10^{-3} mol
Amount of HC*l* in 250 cm^3 of solution = (250/25.0)(2.00 x 10^{-3})
= 0.0200 mol

3. **Determine the volume of HC*l* to be used in the experiment**

Total amount of HC*l* required in the experiment
= 0.02398 + 0.0200 = 0.04398 mol

Volume of 1.00 mol dm^{-3} HC*l* required
= 0.04398/1.00 = 0.04398 dm^3 = 43.98 cm^3 **[1]**

Suitable volume of 1.00 mol dm^{-3} HC*l* to be used: **44.00 cm^3**.

If average titre value of aq NaOH used is assumed to be 25.00 cm^3,
Amount of HC*l* in 25.0 cm^3 of solution = 2.50 x 10^{-3} mol
Amount of HC*l* in 250 cm^3 of solution = 0.0250 mol
Total amount of HC*l* = 0.04898 mol
Volume of 1.00 mol dm^{-3} of HC*l* required = 48.98 cm^3
Suitable volume of 1.00 mol dm^{-3} of HC*l* to be used = 49.00 cm^3

Key points to be included in the procedure:

- ✓ Apparatus used and its capacity: **[1]**
  - analytical / mass balance
  - burette (for measuring HC*l* and titration)
  - pipette
  - 250 cm^3 graduated flask
- ✓ Mass / volume of reagents used **[1]**
  - 1.50 g of impure sample
  - 44.00 cm^3 of 1.00 mol dm^{-3} HC*l*(aq)
  - 25.0 cm^3 of **FA3**
- ✓ Measurements taken **[1]**
  - total mass of weighing bottle and sample
  - mass of emptied weighing bottle
  - titration results
- ✓ Use of funnel to prevent acid spray **[1]**
- ✓ End-point colour change **[1]**
  - Note: Since HC*l* is a strong acid and NaOH is a strong base, this is a strong acid-strong base titration.
- ✓ Repeat titration until at least two consistent results are obtained **[1]**

Procedure:

1. Using an analytical balance, weigh accurately about 1.50 g of the given sample of impure $CaCO_3$ in a clean and dry weighing bottle. Record the total mass of weighing bottle and solid.
2. Using a **burette,** measure 44.00 cm^3 of 1.00 mol dm^{-3} HC*l*(aq) into a **250 cm^3 conical flask**.
3. Add the sample of $CaCO_3$ from the weighing bottle into the conical flask containing the acid carefully and immediately place a clean funnel at the mouth of the conical flask to prevent acid spray.
4. Reweigh the emptied weighing bottle and record its mass.
5. Swirl the conical flask gently. When effervescence has ceased, rinse the filter funnel with some deionised water and allow the washings to drain into the conical flask. *(Note: This helps to ensure that any acid present on the filter funnel will be washed into the conical flask.)*
6. **Transfer** the solution form the conical flask **quantitatively** into a 250 cm^3 graduated flask and **make up the solution to the mark** with deionised water, **using a dropper** when **nearing the mark**. **Stopper** the graduated flask, **shake** well and label this solution **FA 3**.
7. Pipette **25.0 cm^3** of **FA 3** into a **250 cm^3** conical flask and add two drops of methyl orange indicator.
8. Titrate this solution against 0.100 mol dm^{-3} NaOH(aq) placed in a burette.
9. Stop the titration when endpoint is reached i.e. when the solution in the conical flask changes colour from red to orange.
10. Record the titration results.
11. Repeat the titration until at least two consistent results are obtained, i.e. the two titre volumes do not differ by more than 0.10 cm^3.

Commented [TW1]: Reaction vessel

Commented [TW2]: Acid carbonate rxn, need to prevent spray.

Q3(b)

Amount of NaOH used during titration = $(\frac{y}{1000})(0.100) = \frac{y}{10000}$ mol

$NaOH(aq) + HCl(aq) \longrightarrow NaCl(aq) + H_2O(l)$

Amount of HC*l* in 25.0 cm^3 of FA3 = $\frac{y}{10000}$ mol

Amount of HC*l* in 250 cm^3 of FA3 = $(250/25.0)(\frac{y}{10000}) = \frac{y}{1000}$ mol

Total amount of HC*l* added to the $CaCO_3$ sample initially = $(\frac{x}{1000})(1.00) = \frac{x}{1000}$ mol

Amount of HC*l* that reacts with $CaCO_3$ = $(\frac{x}{1000} - \frac{y}{1000})$ mol **[1]**

$CaCO_3(s) + 2HCl(aq) \longrightarrow CaCl_2(aq) + CO_2(g) + H_2O(l)$

Amount of $CaCO_3$ reacted = $\frac{1}{2}(\frac{x}{1000} - \frac{y}{1000})$ mol **[1]**

Molar mass of $CaCO_3$ = 100.1 g mol^{-1}

Mass of $CaCO_3$ in the sample = $(\frac{1}{2})(\frac{x}{1000} - \frac{y}{1000})(100.1) = (\frac{50.05}{1000})(x - y)$ g

Mass of sample used = m g

Percentage purity of $CaCO_3$ in the sample = $(\frac{50.05}{1000})(x - y) \div m \times 100\% = (\frac{5.005}{m})(x - y)$ **% [1]**