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Introduction

Chemistry P525/3 is a practical paper commonly made up of three (3) questions involving quantitative and qualitative analysis.

Qualitative analysis at 'A' level is divide into **inorganic** and **organic** analysis

QUANTITATIVE ANALYSIS

What is Quantitative analysis

Quantitative analysis relies on methods involving accurate measurements of volumes and masses.

Volumetric analysis is a form of quantitative analysis that involves measurement of volumes of reacting solutions. Pipettes, burettes, measuring cylinders and volumetric flasks etc. are used.

Gravimetric analysis: is a form of quantitative analysis that involves measurement of masses of substances. Here use is made of the weighing balance.

Standard solutions

A standard solution is one of whose concentration is accurately known. Units include grams per litre or moles per litre. E.g. 2M sodium hydroxide.

Molarity: is the number of moles of a solute in one litre of a solution. A molar solution is one which contains one mole of a solute in one litre of solution.

Note: 1 litre = 1dm³ = 1000cm³

Primary Standard

A primary standard (or standardizing agent) is a substance of known concentration which is used to prepare a standard solution.

Note: to 'standardize a substance' means 'finding the concentration of the substance in a given solution'

Properties of a good primary standard

- (i) Must be always available in a high state of purity
- (ii) Must be always readily soluble in a given solvent
- (iii) Must be stable in air, that is, should not be hygroscopic, deliquescent or efflorescent and shouldn't decompose at ordinary temperatures.
- (iv) Must have a reasonably high relative molecular mass. This minimizes errors in weighing.
- (v) Must give consistent titre values.
- (vi) Must be fairly cheap to obtain.

Examples of primary standards:

- Anhydrous sodium carbonate
- Pure disodium tetraborate i.e. borax
- Oxalic acid
- Sodium oxalate
- Sodium chloride
- Potassium iodate
- Potassium dichromate etc.

Note:

Sodium hydroxide is not a good primary standard because it is deliquescent and absorbs carbon dioxide from air;

Concentrated sulphuric acid is highly oxidizing, volatile and decomposes on exposure to air. It is also hygroscopic.

Potassium permanganate is not suitable for use as a primary standard because it leaves deposits of manganese(IV) oxide behind and has low solubility.

Titration

A titration is the process of determining the composition of a substance by measuring the volume of one solution needed to react with another solution. At least one of the solutions is a standard solution (its concentration is known). When one solution is an acid and the other a base, the titration is referred to as an acid-base titration. However, if one solution contains a reducing agent and the other solution contains an oxidizing agent, then it is a redox titration.

Indicators

Phenolphthalein and methyl orange indicators are commonly used in acid-base titrations. Both indicators are suitable for titration of a strong base with a strong acid. Phenolphthalein is suitable for strong base with weak acid while methyl-orange is for titration of weak base with strong acid.

Starch indicator is also used in titrations involving iodine (iodometry).

In potassium permanganate titrations, the permanganate solution (purple) itself is the indicator.

Recording results:

Pipette volume is recorded to one decimal place e.g. 25.0, 20.0 or 10.0

Burette readings are strictly recorded to two decimal places e.g. 19.80

Example:

Volume of pipette used = 20.0 cm³

Burette readings:

Final readings (cm ³)	20.10	39.80	19.90
Initial reading (cm ³)	0.00	20.10	0.10
Volume used (cm ³)	20.10	19.70	19.80

These volumes in the table are referred to as titre values. The volumes chosen to calculate average volume used should not differ by more than 0.1cm³.

$$\begin{aligned}\text{Average volume used} &= \frac{19.70+19.80}{2} \\ &= \mathbf{19.75\text{ cm}^3}\end{aligned}$$

These volumes; pipette and burette reading (average), are used in quantitative calculations. Considering the sample records above, 20 cm³ of solution in the pipette reacts with 19.75 cm³ of the solution in the burette.

Worked example

FA1 is a solution of **hydrochloric acid**.

Solid **Z** is **anhydrous sodium carbonate** powder.

You are required to **standardize** FA1 using anhydrous sodium carbonate (Z)

Procedure

Weigh accurately 2.65g of **Z** in beaker, add about 100cm³ of distilled water to dissolve it and transfer to a 250cm³ volumetric flask. Make the solution to the mark with more distilled water. Label the solution FA2.

Pipette 25.0 (or 20.0) cm³ of FA2 into a conical flask, add 2 drops of methyl orange indicator and titrate the resultant mixture with FA1 from the burette.

Repeat the titration until you obtain consistent results. Record your results in the table below.

Results

Volume of pipette used = 25.0 cm³

Burette readings

Final burette reading/cm ³	26.00	36.70	41.10
Initial burette reading/cm ³	0.00	11.50	15.00
Volume of FA1 used/ cm ³	26.00	25.20	25.10

Titre values used to calculate the average volume of FA1 used:

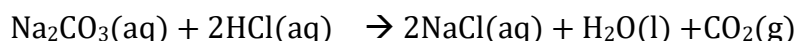
25.20, 25.10 cm³

Average volume of FA1 used

$$\begin{aligned}\text{Average volume used} &= \frac{25.20+25.10}{2} \\ &= \mathbf{25.15\text{ cm}^3}\end{aligned}$$

Questions:

(a) Write equations for the reaction



(b) Determine the molarity of FA2

250cm³ of solution FA2 contain 2.65g of sodium carbonate

1000cm³ of solution FA2 contain $\frac{2.65 \times 1000}{250}$ g of sodium carbonate

$$= 10.6\text{g}$$

$$\text{RFM of Na}_2\text{CO}_3 = (2 \times 23) + 12 + (3 \times 16) = 106$$

106g of sodium carbonate FA2 contain 1mol

$$10.6\text{g of sodium carbonate will contain } \frac{1 \times 10.6}{106} = 0.1 \text{ mol}$$

$$\text{Hence the molarity of FA2} = 0.1 \text{ mol dm}^{-3}$$

(c) Calculate the molarity of FA1

(FA2 is the standard solution used to analyse FA1. 25 cm³ of FA2 reacts with 25.15 cm³ of FA1)

1000cm³ of FA2 contain 0.1 moles

$$25 \text{ cm}^3 \text{ of FA2 contain } \frac{0.1 \times 25}{1000} \text{ moles}$$

Reaction ratio of FA2 : FA1 is 1 : 2

$$\text{Moles of FA1 that reacted} = 2 \times \frac{0.1 \times 25}{1000} \text{ mole (these are moles in 25.15 cm}^3 \text{ of FA1)}$$

$$25.15 \text{ cm}^3 \text{ of FA1 contain } 2 \times \frac{0.1 \times 25}{1000} \text{ moles}$$

$$1000 \text{ cm}^3 \text{ of FA1 contain } 2 \times \frac{0.1 \times 25}{1000} \times \frac{1000}{25.15}$$

$$\text{Molarity of FA1} = 0.199 \text{ mol dm}^{-3}$$

a) Determine the concentration of FA1 in grams per litre.

$$\text{RFM of HCl} = 1 + 35.5 = 36.5$$

$$\text{Therefore 1 mole of HCl will weigh } (36.5 \times 0.199)\text{g} = 7.2635\text{g}$$

$$\text{Concentration of FA1} = 7.2635\text{g/l}$$

ACID BASSE TITRATION

Standardization of sodium hydroxide using hydrochloric acid

You are provided with the following;

FA1, which is a 0.1M hydrochloric acid

M, which is sodium hydroxide

You are required to standardize a solution of sodium hydroxide

Procedure

Weigh accurately about 1.0g of M in a beaker. Add 100cm³ of distilled water and stir to dissolve. Transfer the solution into a 250cm³ volumetric flask and make it to the mark with more water. Label the solution FA2.

Pipette 25.0 (or 20.0) cm³ of FA2 into a conical flask, add 1-2 drops of phenolphthalein indicator and titrate the resultant mixture with FA1 from the burette.

Repeat the titration to obtain consistent readings and record your results in the table below.

Results

Mass of weighing vessel + M =g

Mass of weighing vessel alone =g

Mass of M alone =g

Volume of pipette used =cm

Burette readings

Final readings/ cm ³			
---------------------------------	--	--	--

Initial reading /cm ³			
Volume of FA1 used / cm ³			

Titre values used to calculate the average volume of FA1 used

Average volume of FA1 used

Write equation for the reaction

Calculations

a) Calculate the number of moles of;

(i) FA1 the reacted

(ii) FA2 that reacted and hence the molarity of FA2.

b) Determine the concentration of FA2 in grams per litre.

Standardization of sodium hydroxide using oxalic acid

You are provided with the following:

FA1, which is sodium hydroxide solution.

Z, which is oxalic acid crystals ($\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$).

You are required to standardize the solution hydroxide provided.

Procedure

Weigh accurately about 1.6g of Z in a beaker. Add 100cm³ of distilled water and stir to dissolve. Transfer the solution into a 250cm³ volumetric flask and make it to mark with more water. Label the solution FA2.

Pipette 25.0 (or 20.0) cm³ of FA1 into a conical flask, add 1-2 drops of phenolphthalein indicator and titrate the resultant mixture with FA2 from the burette. Repeat the titration to obtain consistent readings and record your results in the table below.

Results:

Mass of weighing vessel + Z=g

Mass of weighing vessel alone=.....g

Mass of Z alone =.....g

Volume of pipette used =cm³

Burette readings:

Final reading / cm ³			
Initial reading /cm ³			
Volume of FA2 used /cm ³			

Titre values used to calculate the average volume of FA2 used

Average volume of FA2 used

Write equation for the reaction

Calculations

a) Determine the molarity of FA2

.....

.....

.....

b) Calculate:

(i) Moles FA2 that reacted

.....

.....

(ii) Molarity of FA1

.....

.....

.....

.....

c) Determine the concentration of FA1 in grams per litre.

.....

.....

Standardization of hydrochloric acid using sodium tetraborate decahydrate (borax)

You are provided with the following;

FA1, which is a solution containing approximately 0.1M hydrochloric acid.

G; borax crystals

You are required to standardize hydrochloric acid solution.

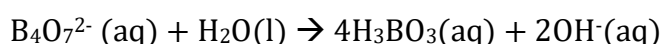
Procedure:

Weigh accurately about 5.0g of G in a beaker. Dissolve in about 100cm³ of distilled water and stir to dissolve. Warm the beaker to speed up dissolution. Cool the solution then transfer the solution into a 250cm³ volumetric flask and make it to mark with more water. Label the solution FA2.

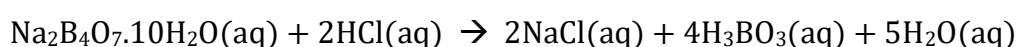
Pipette 25.0 (or 20.0) cm³ of FA2 into a conical flask, add 1-2 drops of methyl orange indicator and titrate the resultant mixture with FA1 from the burette. Repeat the titration to obtain consistent readings and record your results in the table below.

Theory

Borax (Na₂B₄O₇) is a salt of a strong base (NaOH) and weak acid (boric acid H₃BO₃). It undergoes hydrolysis in aqueous solution to form hydroxide ions.



These OH⁻ ions make the solution alkaline and hence react with mineral acids.



Results:

Mass of weighing vessel + G =g

Mass of weighing vessel alone =g

Mass of G alone =g

Volume of pipette used =cm

Burette readings

Final readings/ cm ³			
Initial reading /cm ³			
Volume of FA1 used / cm ³			

Average volume of FA1 used

.....

Calculate:

a) Molar concentration of FA2

.....

b) Hence determine the concentration of FA1 in gams per litre.

.....

Determination of number of molecules of water of crystallization of oxalic acid

You are provided with the following

FA1, which is 0.1M sodium hydroxide

Solid A, which is oxalic acid crystals H₂C₂O₄·nH₂O

Produce:

Weigh accurately about 1.60g of A in a beaker and dissolve in about 100cm³ of distilled water. Transfer to a 250cm³ volumetric flask and make up to the mark.

Label the solution FA2.

Pipette 25.0 (or 20.0) cm³ of FA2 into a conical flask, add 2 drops of phenolphthalein indicator and titrate the resultant mixture with FA1 from the burette.

Repeat the titration to obtain consistent readings and record your results in the table below.

Results:

Mass of weighing vessel + A =g

Mass of weighing vessel alone =g

Mass of A alone =g

Volume of pipette used =cm

Burette readings

Final readings/ cm ³			
Initial reading /cm ³			
Volume of FA1 used / cm ³			

Titrate values used to calculate the average volume of FA1 used

Average volume of FA1 used

Write equation for the reaction between sodium hydroxide and oxalic acid.

Calculations:

a) Calculate:

(i) The molar concentration of FA2

(ii) Molar mass of A and hence the value of n in $H_2C_2O_4 \cdot nH_2O$

Back Titration

Back titration involves analysing the composition of a substance that is insoluble in water by reacting it with excess reagent and then titrating the unreacted reagent with a standard solution. E.g. calcium carbonate which is insoluble in water is determined by treating it with a known amount standard hydrochloric acid (say 2.0M) and then the unreacted excess acid titrated with a standard base.

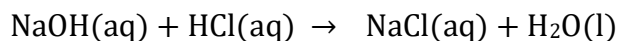
Worked example

50cm³ of 1.0M hydrochloric acid was added to 0.78g of a divalent metal oxide MO. The resultant solution was transferred to a 250cm³ volumetric flask and topped to the mark with water.

25cm³ of this solution required 21.10cm³ of 0.1M sodium hydroxide on titration for complete neutralization.

a) Calculate:

i) Moles of sodium hydroxide that reacted



100cm³ of solution contain 0.1 moles of NaOH

21.1cm³ would contain $\frac{21.1 \times 0.1}{1000}$ moles = 0.00211 of NaOH

ii) Moles of excess hydrochloric acid that reacted with sodium hydroxide

From the equation, NaOH: HCl = 1:1

Moles of excess hydrochloric acid that reacted with sodium hydroxide

$$= 1 \times 0.00211$$

$$= 0.00211 \text{ moles}$$

iii) Moles of hydrochloric acid that reacted with MO

25cm³ of solution contained 0.00211 moles of excess acid

250cm³ of solution contain $\frac{250 \times 0.00211}{25} = 0.0211$ moles of excess acid

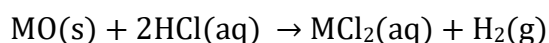
But 1000cm³ of the original acid contained 1.0mole

Hence 50cm³ of the acid contain $\frac{50 \times 1}{1000}$ moles = 0.05moles

Hence moles of acid that reacted with MO = 0.05 – 0.0211 = 0.0289 moles

iv) Moles MO that reacted with the acid

Equation of reaction



MO : HCl = 1:2

Hence moles of MO that reacted with the acid = $\frac{1}{2} \times 0.0289 = 0.01445$ moles

b) Hence determine the molar mass of MO and hence the atomic mass of M in MO

0.01445 moles of MO weighed 0.78g

Hence 1 moles of MO would weigh $\frac{0.78 \times 1}{0.01445} = 53.98\text{g}$

Therefore, the RFM of MO = 53.98

Hence MO = 53.98

$$\text{M} + 16 = 53.98$$

$$\text{M} = 37.98$$

Determination of relative atomic mass of an element in a carbonate

You are provided with the following

FA1, which is 1.0M hydrochloric acid solution

FA2, which is 0.1M sodium hydroxide

Solid Y which is a metal carbonate MCO_3

You are required to determine the atomic mass of metal atom M.

Procedure

Weigh accurately 1.5g of MCO_3 in a beaker, add about 20cm^3 of distilled water to cover it, the 50cm^3 of FA1 and stir until effervescence stops. Transfer it into a 250cm^3 volumetric flask and make up to the mark. Label this solution FA3. Pipette 25.0 (or 20.0) cm^3 of FA3 into a conical flask, add 2 drops of methyl orange indicator and titrate the resultant mixture with FA2 from the burette.

Repeat the titration to obtain consistent readings and record your results in the table below.

Results

Mass of weighing vessel + MCO_3 =g

Mass of weighing vessel alone =g

Mass of MCO_3 =g

Volume of pipette used = cm^3

Burette readings:

Final reading/ cm^3			
Initial reading/ cm^3			
Volume of FA2 used/ cm^3			

Titrate values used to calculate the average volume of sodium hydroxide used

Average volume of sodium hydroxide used

a) Calculate:

i) Moles of excess acid

.....

.....

.....

.....

ii) Moles of acid that reacted with MCO_3 .

.....

.....

iii) Atomic mass of symbol M

.....

.....

.....

Estimation of ammonia in ammonium chloride

You are provided with the following
 FA1, which is 1.0M sodium hydroxide
 FA2, which is 0.1M hydrochloric acid.
 Solid B, which is ammonium chloride
 You are required to determine the percentage of ammonium chloride.

Procedure

Weigh out accurately 5.0g of a sample of ammonium chloride in a 500ml beaker. Add 100 cm³ of 2.0M sodium hydroxide and boil the mixture until ammonia has ceased to evolve (test with red litmus)

Cool the solution, transfer it into a 250cm³ volumetric flask and make it to the mark with distilled water. Label this solution FA3.

Pipette 25.0 (or 20.0) cm³ of FA3 into a conical flask, add 1- 2 drops of phenolphthalein indicator and titrate the resultant mixture with FA2 from the burette.

Repeat the titration to obtain consistent readings and record your results in the table below.

Results

Mass of weighing vessel + solid B =.....g

Mass of weighing vessel alone =.....g

Mass of solid B alone =.....g

Volume of pipette used =.....cm³

Burette readings:

Final reading/cm ³			
Initial reading/cm ³			
Volume of HCl used/cm ³			

Titre values used to calculate the average volume of sodium hydroxide used

Average volume of hydrochloric acid used

b) Calculate the:

i) Moles of excess sodium hydroxide.

.....

.....

.....

.....

.....

ii) Moles of sodium hydroxide that reacted with ammonium chloride.

.....

.....

.....

(b) Hence determine the percentage of ammonia in ammonium chloride.

.....

.....

.....

.....

Determination of the number of molecules of water of crystallization in a hydrated acid

You are provided with the following
 FA1, which is 1.0M sodium hydroxide
 FA2, which is 0.1M hydrochloric acid.
 N, which is a hydrated acid $H_2Y.nH_2O$. ($Y=88$)
 You are required to determine n, in $H_2Y.nH_2O$.

Procedure

Weigh out accurately 4.7g of N in a 500ml beaker. add 100cm^3 of FA1 and shake to dissolve. Transfer it into a 250ml volumetric flask and make up to the mark with distilled water. Label this solution FA3.

Pipette 25.0 (or 20.0) cm^3 of FA3 into a conical flask, add 2 drops of phenolphthalein indicator and titrate the resultant mixture with FA2 from the burette.

Repeat the titration to obtain consistent readings and record your results in the table below.

Results

Mass of weighing vessel + N =g

Mass of weighing vessel alone =g

Mass of solid N alone =g

Volume of pipette used = cm^3

Burette readings:

Final reading/ cm^3			
Initial reading/ cm^3			
Volume of FA2 used/ cm^3			

Titre values used to calculate the average volume FA2 used

Average volume of FA2 used

- c) Calculate the:
 iii) Moles of excess FA1.

- iv) Moles of FA1 that reacted with N.

- b) Hence determine the value of n in $H_2Y.nH_2O$.

DOUBLE INDICATOR TITRATION

Introduction:

A double indicator titration is a technique used for analysing the composition of a mixture with the use of two different indicators.

It can be done in two ways:

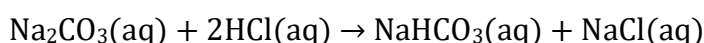
- a) Two separate titrations (non-continuous method) are done independently with the indicators.
- b) One indicator (usually phenolphthalein) is added first, and at end point, the second indicator (usually methyl-orange) is added. Then titration is done to completion (continuous method).

The common mixtures considered include;

- i) Sodium carbonate with sodium hydrogen carbonate.
- ii) Sodium carbonate with sodium hydroxide.
- iii) Sodium hydrogen carbonate with sodium hydroxide.

This method of analysis is based on the fact that the reaction of Na_2CO_3 with acids is a two-step reaction.

Step 1.



Step 2.



Second to that, the indicator (methyl orange and phenolphthalein) responds differently to these steps.

Titration with phenolphthalein

When phenolphthalein is used in the titration of sodium carbonate, end point is indicated at half neutralisation (at end of step 1). This implies that the actual volume of acid required for complete reaction would be twice the observed volume (step1 + step2). E.g. if the volume of sulphuric acid required to reach end point for a reaction with sodium carbonate using phenolphthalein indicator was 12.40cm^3 , the complete reaction requires 24.8cm^3 of acid (i.e. 2×12.4)

Note: step 2 is not detected by phenolphthalein. Action of the indicator is affected by carbon dioxide. It also means that if a solution contains NaHCO_3 , phenolphthalein will not indicate end point.

Titration with methyl orange

During a titration with an acid, the carbonate reacts completely to give a salt, water and carbon dioxide gas. Any hydrogen carbonate also reacts completely.

Worked example

Analysis of mixture of sodium carbonate and sodium hydroxide.

You are provided with the following:

ZA1, which is 0.1M hydrochloric acid.

ZA2, which is a solution containing a mixture of sodium carbonate with sodium hydroxide.

You are required to determine the concentration of each of the components in gdm^3 .

Method 1 (continuous titration)

Procedure

- Pipette 25cm³(or 20cm³) of ZA2 into a clean conical flask, add 2 drops of phenolphthalein indicator and titrate with ZA1 from the burette. Record the results in table 1.
- Add 2-3 drops of methyl orange to the contents of the conical flask from (a) and continue the titration. Record the results in table 2.
- Repeat (a) and (b) until you obtain consistent results.

Results

Volume of pipette used: 25.0cm³

Burette reading

Table 1 (using phenolphthalein)

Final burette reading /cm ³	23.70	23.40	27.30
Initial burette reading /cm ³	0.20	0.00	3.90
Volume of ZA1 used /cm ³	23.50	23.40	23.40

Average volume of ZA1 used: =23.40cm³

Table 2 (using methyl-orange)

Final burette reading /cm ³	34.40	35.50	37.90
Initial burette reading /cm ³	23.70	24.40	27.30
Volume of ZA1 used /cm ³	10.70	10.60	10.60

Average volume of ZA1 used: $\frac{10.7+10.6}{2}$ =10.55cm³

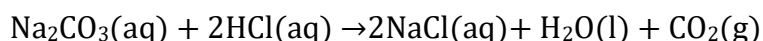
Calculate

- Concentration of Na₂CO₃ in gdm⁻³

Solution

Volume of HCl that reacted (complete neutralisation) = 10.55x2=21.10cm³

$$\text{Moles of HCl that reacted} = \frac{21.1 \times 0.1}{1000} = 0.00211 \text{ mol}$$



$$\text{Moles of Na}_2\text{CO}_3 \text{ that reacted} = \frac{0.00211}{2} = 0.001055 \text{ mol}$$

$$\text{Moles of Na}_2\text{CO}_3 \text{ in } 25.0\text{cm}^3 = \frac{0.00211}{2} = 0.001055 \text{ mol}$$

$$\text{Moles of Na}_2\text{CO}_3 \text{ in } 1000\text{cm}^3 = \frac{0.001055 \times 1000}{25} = 0.0422 \text{ mol}$$

$$\text{RFM of Na}_2\text{CO}_3 = (23 \times 2) + 12 + (16 \times 3) = 106$$

1 mole contains 106g of Na₂CO₃

$$0.0422 \text{ mol contain } 106 \times 0.0422 = 4.47 \text{ g}$$

Concentration of Na_2CO_3 in ZA2 = 4.47gdm^{-3}

b) Concentration of NaOH in gdm^{-3}

Solution

Volume of HCl that reacted (complete neutralisation) = $23.40 - 10.55 = 12.85\text{cm}^3$

Moles of HCL that reacted = $\frac{12.85 \times 0.1}{1000}$

= 0.001285 mol

$\text{NaOH(aq)} + \text{HCl(aq)} \rightarrow \text{NaCl(aq)} + \text{H}_2\text{O(l)}$

Reaction ratio of NaOH: HCl = 1: 1

Moles of NaOH that reacted = 0.001285mol

Moles of NaOH in $25.0\text{cm}^3 = 0.001285\text{mol}$

Moles of NaOH in $1000\text{cm}^3 = \frac{0.001285 \times 1000}{25} = 0.0514\text{ mol}$

RFM of NaOH = $23 + 16 + 1 = 40$

1 mole contain 40g of NaOH

0.0514 mol contain $40 \times 0.0514 = 2.056\text{g}$

Concentration of NaOH in ZA2 = 2.056gdm^{-3}

c) Percentage of sodium carbonate in the mixture.

$$\frac{4.47}{4.47 + 2.05} \times 100\% = 68.5\%$$

Method II (Non continuous method)

Procedure

- Pipette 25cm^3 (or 20cm^3) of ZA2 into a clean conical flask, and 2 drops of phenolphthalein indicator and titrate with ZA1 from the burette. Repeat the titration to obtain consistent results. Record the results in the table 1.
- Pipette 25cm^3 (or 20cm^3) of ZA2 into a clean conical flask, and 2 drops of methyl-orange indicator and titrate with ZA1 from the burette. Repeat the titration to obtain consistent results. Record the results in tables.

Results

Volume of pipette used: 25.0cm^3

Burette readings

Table 1 (using phenolphthalein indicator)

Final burette reading / cm^3	23.70	23.40	27.30
Initial burette reading / cm^3	0.20	0.00	3.90
Volume of ZA1 used / cm^3	23.50	23.40	23.40

Average volume of ZA1 used = $\frac{23.4 + 23.4}{2} = 23.40\text{ cm}^3$

Table 2 (using methyl-orange indicator)

Final burette reading / cm^3	34.40	35.50	37.90
Initial burette reading / cm^3	0.00	1.00	3.80
Volume of ZA1 used / cm^3	34.20	34.00	34.00

Average volume of ZA1 used: $\frac{34.4+34.4}{2} = 34.00 \text{ cm}^3$

Calculate

d) Concentration of Na_2CO_3 in gdm^{-3}

Solution

Volume of HCl that reacted (complete neutralisation) = $2(34.00-23.40) = 21.20 \text{ cm}^3$

Moles of HCl that reacted = $\frac{21.2 \times 0.1}{1000} = 0.00212 \text{ mol}$

$\text{Na}_2\text{CO}_3(\text{aq}) + 2\text{HCl}(\text{aq}) \rightarrow 2\text{NaCl}(\text{aq}) + \text{H}_2\text{O}(\text{l}) + \text{CO}_2(\text{g})$

Moles of Na_2CO_3 that reacted = $\frac{0.00212}{2} = 0.00106 \text{ mol}$

Moles of Na_2CO_3 in $25.0 \text{ cm}^3 = 0.00106 \text{ mol}$

Moles of Na_2CO_3 in $1000 \text{ cm}^3 = \frac{0.00106 \times 1000}{25} = 0.0424 \text{ mol}$

RFM of $\text{Na}_2\text{CO}_3 = (23 \times 2) + 12 + (16 \times 3) = 106$

1 mole contains 106g of Na_2CO_3

0.0424 mol contain $106 \times 0.0424 = 4.494 \text{ g}$

Concentration of Na_2CO_3 in ZA2 = 4.494 gdm^{-3}

e) Concentration of NaOH in gdm^{-3}

Solution

Volume of HCl that reacted (complete neutralisation) = $23.40 - 10.6 = 12.80 \text{ cm}^3$

Moles of HCl that reacted = $\frac{12.8 \times 0.1}{1000} = 0.00128 \text{ mol}$

$\text{NaOH}(\text{aq}) + \text{HCl}(\text{aq}) \rightarrow \text{NaCl}(\text{aq}) + \text{H}_2\text{O}(\text{l})$

Moles of NaOH that reacted = 0.00128 mol

Moles of NaOH in $25.0 \text{ cm}^3 = 0.001285 \text{ mol}$

Moles of NaOH in $1000 \text{ cm}^3 = \frac{0.001285 \times 1000}{25} = 0.0512 \text{ mol}$

RFM of NaOH = $23 + 16 + 1 = 40$

1 mole contain 40g of NaOH

0.0514 mol contain $40 \times 0.0512 = 2.048 \text{ g}$

Concentration of NaOH in ZA2 = 2.048 gdm^{-3}

f) Percentage of sodium carbonate in the mixture.

$$\frac{4.494}{4.494 + 2.048} \times 100\% = 68.3\%$$

Exercise

Analysis of a mixture of sodium carbonate with sodium hydrogen carbonate.

You are provided with the following:

FA1, which is a solution containing sodium carbonate and sodium hydrogen carbonate.

FA2, which is a 0.1M hydrochloric acid

You are required to determine the concentration in grams per litre of each component of FA1.

Procedure

a) Pipette 25.0 or 20.0 cm^3 of FA1 into a conical flask and add 2 drops of phenolphthalein indicator. Titrate the mixture with FA2 until it turns red. Record the results in table II.

b) The add 2 drops of methyl orange indicator to the resultant solution in (a) and continue the titration with FA2 until it turns red. Record the results in table II.

c) Repeat procedures (a) and (b) to obtain consistent readings.

Volume of pipette used =cm³

Burette readings:

Table 1

Final reading/cm ³			
Initial reading/cm ³			
Volume of FA2 used/cm ³			

Average volume

.....

Table II

Final reading/cm ³			
Initial reading/cm ³			
Volume of FA2 used/cm ³			

Average volume of FA2 used

.....

Questions:

a) Determine the volume of FA2 that reacted with;

(i) Sodium carbonate.

.....

(ii) Sodium hydrogen carbonate.

.....

b) Hence calculate the concentration in grams per litre of:

(i) Sodium carbonate

.....

(ii) Sodium hydrogen carbonate

.....

Analysis of a mixture of sodium carbonate and sodium hydroxide.

You are provided with the following:

FA1, which is a solution containing sodium carbonate and sodium hydrogen carbonate.

FA2, which is a 0.1M hydrochloric acid

You are required to determine the percentage of sodium hydroxide in mixture.

Procedure

Part 1 below

Pipette 25.0 / 20.0cm³ of FA1 into a conical flask, add 1-2 drops of phenolphthalein indicator and titrate the resultant mixture with FA2 from the burette. Repeat the titration to obtain consistent readings and record your results in table 1 below.

Volume of pipette used = cm³

Table1

Final burette reading/ cm ³			
Initial burette reading/ cm ³			
Volume of FA2 used / cm ³			

Average volume of FA2

.....

Part II

Pipette 25.0/20.0 cm³ of FA1 into a conical flask, add 1-2 drops of methyl-orange indicator and titrate the resultant mixture with FA2 from the burette.

Repeat the titration to obtain consistent readings and record your results in the table II below.

Volume of pipette used cm³

Table II

Final burette reading/ cm ³			
Initial burette reading/ cm ³			
Volume of FA2 used / cm ³			

Average volume of FA2

.....

- a) What volume of FA2 reacted with:
i) Sodium carbonate?

.....

- ii) Sodium hydroxide?

.....

- b) Determine the concentration in grams per litre of:
i) Sodium carbonate

.....
.....
.....

.....

 ii) Sodium bicarbonate

c) Hence calculate the percentage of sodium hydroxide in FA1

Analysis of a mixture of sodium hydrogen carbonate and sodium hydroxide.

You are provided with the following:

FA1, which is a solution containing a mixture of sodium hydrogen carbonate and sodium hydroxide.

ZA2, which is a 0.05M sulphuric acid.

You are required to determine the composition of FA1

Procedure

- Pipette 25cm³ or 20cm³ of FA1 into a conical flask, add 2-3 drops of phenolphthalein indicator and titrate with FA2 until end point. Record the results in table 1.
- Add 2-3 drops of methyl orange to the contents of the conical flask from (a) and continue the titration. Record the results in table II.
- Repeat (a) and (b) until you obtain consistent results.

Volume of pipette used =cm³

Table I

Final burette reading /cm ³			
Initial burette reading /cm ³			
Volume of FA2 used /cm ³			

Average volume of FA2 used

Table II

Final burette reading /cm ³			
Initial burette reading /cm ³			

Volume of FA2 used /cm ³		
-------------------------------------	--	--

Average volume of FA2 used

Questions:

a) Determine the volume of Fa2 that reacted with:

i) Sodium hydrogen carbonate

ii) Sodium hydroxide

b) Hence calculate the concentration in grams per litre of:

(i) Sodium hydrogen carbonate

(ii) Sodium hydroxide

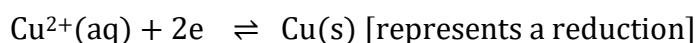
REDOX TITRATION

Titration based on oxidation and reduction reactions are called redox titrations.

These reactions proceed with the transfer of electrons amongst the reacting ions in aqueous solutions.

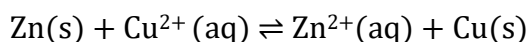
A redox reaction is one in which reduction and oxidation reaction take place simultaneously.

Reduction is electron gain. In a reduction reaction, addition of electron(s) occurs. Oxidation is electron loss; thus, an oxidation reaction is one where there is electron loss or removal.



The two equations above are also referred to as reduction and oxidation half equations respectively.

When the two equations above are also referred to as a reduction and oxidation is a redox equation. i.e.

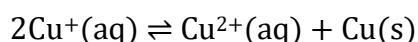


A titration, therefore, in which zinc is oxidized by copper to zinc ions and copper(II) ions reduced to copper, is a redox titration.

An oxidizing agent is a substance which increases the oxidation number of another substance by removing electrons from it.

A reducing agent is a substance which decreases the oxidation number of another substance by adding electrons to it.

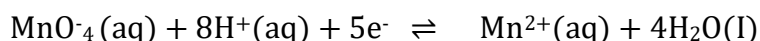
Disproportionation is a redox reaction in which the same species is both oxidized and reduced e.g. Cu^+ ion disproportionate as;



Potassium Permanganate Titrations

Potassium permanganate (also called potassium manganate(VII)) is a strong oxidizing agent which is soluble in water and its aqueous solution is purple. The solution contains the permanganate or manganate (VII) ion; MnO_4^- . Under acidic conditions, potassium permanganate reacts with reducing agents during which manganese is reduced from manganese (vii) ion (purple) to manganese (ii) ion colourless). It is acidified using sulphuric acid (about 2M).

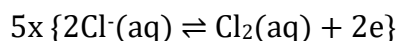
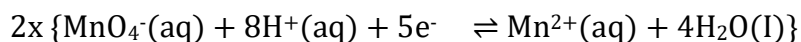
The ionic half equation for reduction of the manganese (vii) ion.



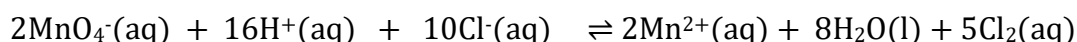
Note:

Potassium permanganate is not acidified using hydrochloric acid because it reacts with hydrochloric acid to form chlorine, which itself is an oxidizing agent. Any reaction of the potassium permanganate with a reducing agent will be affected by chlorine competing for it.

Equations



Over all equation



Nitric acid is not used to acidify it either because nitric acid itself is highly oxidizing as the permanganate. Both species will react with the reducing agent.

Potassium permanganate is not a good primary standard:

- (i) It is impure i.e. contaminated with manganese(IV) oxide. Its solution should be filtered first
- (ii) In presence of an alkaline solution, it gets reduced to a dark brown manganese dioxide.
- (iii) It is so powerful that it cannot be acidified with hydrochloric acid since it oxidizes the Cl^- ions of the acid to chlorine which is itself an oxidizing agent.

- (iv) It is slowly reduced by ordinary distilled water to manganese (iv) oxide especially in presence of an acid or strong sun light.

Equation

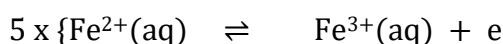


Reducing agents used with potassium permanganate in volumetric analysis

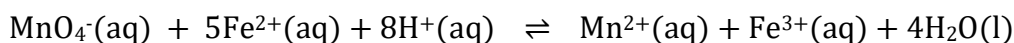
- (i) Iron (ii) salts e.g. ferrous sulphate, ammonium ferrous sulphate.

Under acidic conditions, iron (ii) reduces MnO_4^- (purple) to Mn^{2+} (colourless) and is itself oxidized to iron (iii).

Half equations



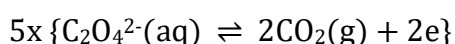
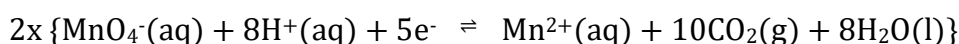
Over all equation



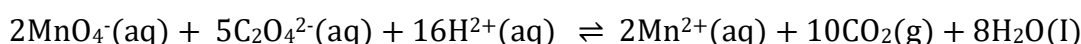
- (ii) Oxalates e.g. oxalic acid, sodium oxalate

The oxalate ion is a strong reducing agent. It reduces MnO_4^- ions to Mn^{2+} ion and is itself oxidized to CO_2 gas.

Half equations



Over all equation

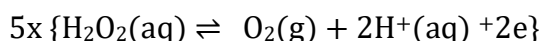
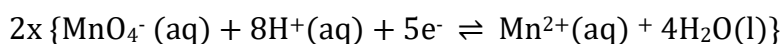


The reaction is carried out at 60-80°C.

As the reaction proceeds, the permanganate is decolourised and a slight excess of it produces a pink colouration at end point.

- (iii) Under acidic conditions, hydrogen peroxide reduces MnO_4^- (purple) to Mn^{2+} (colourless) and itself oxidized to oxygen gas i.e. effervescence of a colourless gas that rekindles a glowing splint.

Half equations

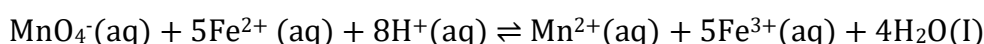


Worked example

40cm³ of 0.02M potassium permanganate acidified with sulphuric acid required exactly 40 cm³ of iron (ii) sulphate ($\text{FeSO}_4 \cdot x\text{H}_2\text{O}$) solution containing 27.8 grams per litre for complete reaction. Determine the molarity of iron (ii) sulphate and the value of x.

Solution

Equation of reaction



1000cm³ of the solution contained 0.02 moles of potassium permanganate

Hence 40cm³ of solution would contain $\frac{40 \times 0.02}{1000} = 0.0008\text{mol}$

From the equation, $\text{MnO}_4^- : \text{Fe}^{2+} = 1.5$

Moles of $\text{Fe}^{2+} = 5 \times 0.0008 = 0.004$

Hence 40cm^3 of solution contained 0.004 moles of Fe^{2+}

Therefore, 1000cm^3 of the solution would contain $\frac{40 \times 0.02}{1000} = 0.1\text{M}$

0.1 moles of iron (ii) sulphate weigh 27.8g

Hence 1 mole weighs $\frac{27.8}{0.1} \times 1 \text{ g} = 278\text{g}$

RFM ($\text{FeSO}_4 \cdot x\text{H}_2\text{O}$) = 278.

$56 + 32 + 64 + 18x = 278$

$x = 7$

Standardization of potassium permanganate using iron (II) salt

You are provided with the following:

FA1, which is approximately 0.02M potassium permanganate

K, which is ammonium ferrous sulphate ($\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$)

2M sulphuric acid.

Procedure:

Weigh accurately 9.80g of K into a beaker, add about 150cm^3 of 2M sulphuric acid and stir to dissolve. Transfer the solution to a 250cm^3 volumetric flask and make it to the mark with distilled water. Label the solution FA3.

Pipette 25.0 or 20.0cm^3 of FA3 into a clean conical flask, and 15cm^3 of 2M sulphuric acid and titrate the mixture with FA1 until the first permanent pink colouration appears. Repeat the procedure to obtain consistent readings and record your results below.

Results:

Mass of weighing vessel + K = g

Mass of weighing vessel alone = g

Mass of K alone = g

Volume of pipette used = g

Burette readings

Final burette reading/ cm^3			
Initial burette reading/ cm^3			
Volume of FA1 used / cm^3			

Titre values used to calculate the average volume FA1 used

.....

Average volume of FA1 used

.....

Write the ionic equation for the reactions which occurred

.....

Questions

(Fe=56, N= 14, S =32, O =16, H =1, Mn =55, K =39)

Calculate the;

a) Molarity of FA3.

.....

.....

.....

b) Concentration in grams per litre of FA1

.....

.....

.....

.....

.....

.....

.....

.....

.....

Standardization of potassium permanganate using sodium oxalate

You are provided with:

FA1, which is a solution containing approximately 2.38 grams per litre of manganate (vii) ions

S, which is sodium oxalate

2M sulphuric acid.

You are required to standardize FA1.

Procedure:

Weigh accurately 1.7g of S into a clean beaker, add 50cm³ of distilled water and shake to dissolve. Transfer the solution to a 250ml volumetric flask and make up to the mark. Label the solution FA3.

Pipette 25.0 or 20.0cm³ of FA3 into a clean conical flask, add an equal volume of 2M sulphuric acid and heat the mixture to 80°C.

Titrate the hot solution immediately with FA1 until a permanent pink coloration appears. Repeat the procedure to obtain consistent readings and record your results below.

Results:

Mass of weighing vessel t S =.....g

Mass of weighing vessel alone =.....g

Mass of S alone =.....g

Volume of pipette used =.....cm³

Burette readings

Final burette reading/cm ³			
Initial burette reading/cm ³			

Volume of FA1 used /cm ³		
-------------------------------------	--	--

Average volume of FA1 used

Write the ionic equation for the reactions which occurred

Questions

Calculate the;

a) Molarity of FA3.

b) The molarity of FA1 and its concentration in grams per litre

Determination of the number of molecules of water of crystallization in iron(ii) sulphate

You are provided with the following:

FA1, which is approximately 0.02M potassium permanganate

F, which is iron (II) sulphate crystals; FeSO₄.nH₂O)

2M sulphuric acid.

You are required to determine the value of n

Procedure:

Weigh accurately 7.0g of F into a beaker, add 100cm³ of 2M sulphuric acid. stir to dissolve and Transfer the solution into a 250cm³ volumetric flask. add 15cm³ of 2M sulphuric acid and titrate the mixture with FA1 until the first permanent pink colouration appears. Make it to the mark with distilled water. Label the solution FA3.

Pipette 25.0 or 20.0cm³ of FA3 into a clean conical flask, and 15cm³ of 2M sulphuric acid and titrate the mixture with FA1 until the first permanent pink colouration appears. Repeat the procedure to obtain consistent readings and record your results below.

Results:

Mass of weighing vessel t F =.....g

Mass of weighing vessel alone =.....g

Mass of F alone =.....g

Volume of pipette used =.....g

Burette readings

Final burette reading/cm ³			
Initial burette reading/cm ³			
Volume of FA1 used /cm ³			

Titre values used to calculate the average volume FA1 used

.....

Average volume of FA1 used

.....

Write the ionic equation for the reactions which occurred

.....

Questions

a) Calculate the;

i) molarity of FA1 that reacted.

.....

(ii) molarity of FA2

.....

b) Determine the value of n

.....

Estimation of percentage of oxalic acid in a mixture with an oxalate

You are provided with the following:

FA1, which is a solution of a mixture of oxalic acid and sodium oxalate

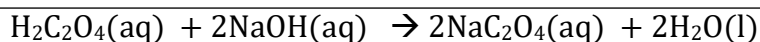
FA2, which is 0.02M potassium permanganate

FA3, which is 0.1M sodium hydroxide

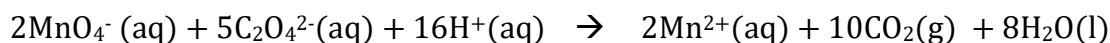
2M sulphuric acid.

Theory

Sodium hydroxide reacts with oxalic acid as shown below



While potassium permanganate, oxidizes oxalate ions as shown below



Procedure:

Part 1

Pipette 25.0 or 20.0cm³ of FA1 into a conical flask, add 1 – 2 drops of phenolphthalein indicator and titrate the resultant mixture with FA3 from the burette.

Repeat the titration to obtain consistent readings and record your results in the table below.

Results:

Volume of pipette used =.....cm³

Mass of weighing vessel t F =.....g

Burette readings

Final burette reading/cm ³			
Initial burette reading/cm ³			
Volume of FA3 used /cm ³			

Titre values used to calculate the average volume of FA3 used

.....

Average volume of FA3 used

.....

a) Calculate

i) moles of FA3 that reacted.

.....

iii) the molar concentration of oxalic acid

.....

b) Determine the concentration of oxalic acid in grams per litre

.....

Part II

Pipette 25.0 or 20.0cm³ of FA1 into a conical flask, add an equal volume of 2M sulphuric acid and then heat the mixture to 80°C.

Titrate the hot solution immediately with FA2 until a permanent pink coloration appears.

Repeat the procedure to obtain consistent readings and record your results in the table below.

Results:

Volume of pipette used = cm³

Burette readings

Final burette reading/cm ³			
Initial burette reading/cm ³			
Volume of FA2 used /cm ³			

Titre values used to calculate the average volume of FA2 used

.....

Average volume of FA2 used

.....

- a) Calculate
i) moles of FA2 that reacted.

.....
.....
.....

- ii) the total molar concentration of oxalate acid

.....
.....
.....
.....
.....
.....
.....
.....
.....
.....

- b) the molarity of sodium oxalate

.....
.....

- c) calculate the percentage of oxalic acid in FA1.

.....
.....
.....
.....

Estimation of volume strength of hydrogen peroxide

You are provided with the following:

FA1, which is hydrogen peroxide

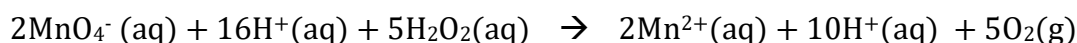
FA2, which is 0.02M potassium permanganate

1.0M sulphuric acid.

Theory

Volume strength is the volume of oxygen liberated by a unit volume of hydrogen peroxide at stp.

In presence of acid, hydrogen peroxide is oxidized by permanganate ions as follows:



Procedure

Transfer carefully 25cm³ of FA1 using a measuring cylinder into a 250cm³ volumetric flask and dilute it to the mark with distilled water.

Pipette 25cm³ of this solution into a conical flask, add 100cm³ of water and 10cm³ of 1.0M sulphuric acid. Titrate the mixture with FA2.

Repeat the titration to obtain consistent titre values and record your results in the table below.

Results:

Volume of pipette used = cm³

Burette readings

Final burette reading/cm ³			
Initial burette reading/cm ³			
Volume of FA3 used /cm ³			

Average volume of FA3 used

.....

a) Calculate

i) moles of FA2 that reacted.

.....

ii) moles of hydrogen peroxide that reacted.

.....

iii) concentration of hydrogen peroxide in the original solution in gdm⁻³

.....

b) Determine the volume strength of the original hydrogen peroxide in litres

.....

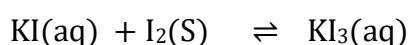
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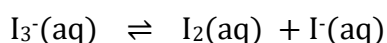
THIOSULPHATE TITRATIONS (iodimetry and iodometry)

The reduction of free iodine to iodide ions and oxidation of iodide ions to free iodine occur in iodometric and iodometric titrations.

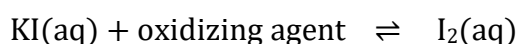
In iodimetry titrations, free iodine is used. Iodine has very low solubility in water because it is non polar molecule and therefore does not form hydrogen bonds with water molecules. It however readily dissolves in aqueous potassium iodide solution due to formation of a soluble triiodide complex



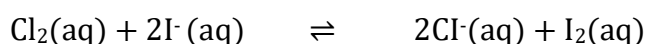
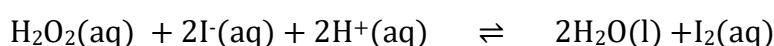
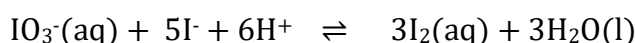
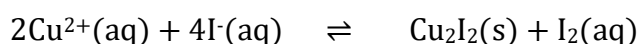
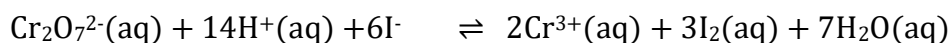
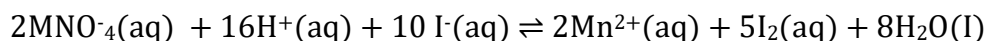
The triiodide ion readily releases iodine into solution.



In iodometric titrations, an oxidizing agent is allowed to react in neutral medium or in acidic medium with excess of KI to liberate free iodine.

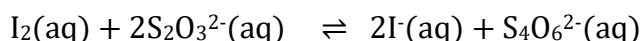


Example:



Note: Chlorine is available in jik

The free iodine formed is titrated against a standard reducing agent usually with sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3$)



These titrations involve the use of a standard thiosulphate solution and therefore referred to as thiosulphate titrations

The commonest source of the thiosulphate is sodium thiosulphate-5-water ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$). it is soluble in water and its aqueous solution is colourless.

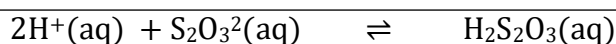
Sodium thiosulphate is not used as a primary standard because:

- (i) Its aqueous solution absorbs carbon dioxide when exposed to air and becomes cloudy/turbid.

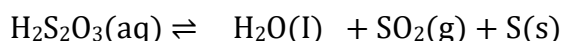
The absorbed carbon dioxide dissolves in water to form carbonic acid, which reacts with the thiosulphate to form thiosulphuric acid.



Then



The thiosulphuric acid is unstable and decomposes immediately to sulphur dioxide and sulphur which makes the solution cloudy.



(ii) Sodium thiosulphate has variable water content.

Detection of end point

End point of thiosulphate titration with iodine is detected using freshly prepared starch indicator added when the iodine solution turns pale yellow (i.e. towards the end point). The solution will then turn blue and at the end point, it turns colourless.

Standardization of iodine solution using standard sodium thiosulphate solution

You are provided with the following;

FA1, which is a solution made by dissolving 12.4g of sodium thiosulphate-5-water ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) in 1 litre of distilled water.

FA2, which is W g of iodine in 40g of potassium iodide dissolve to make 1 litre of solution

Procedure:

Pipette 25.0 (or 20.0) cm^3 of FA2 into a clean conical flask and titrate with FA1 from the burette until the solution turns pale yellow. Add 2 cm^3 of starch solution and continue the titration till endpoint.

Repeat the procedure to obtain consistent results.

Record your results in the table below.

Results: capacity of pipette used: cm^3

Burette readings:

Final reading (cm^3)			
Initial reading (cm^3)			
Volume of FA1 used (cm^3)			

Titre values used to calculate average: cm^3

Average volume of FA1 used: cm^3

Write an ionic equation for the reaction:

.....

a) Calculate:

i) Moles of FA1 that reacted:

.....

ii) Moles of iodine that reacted:

.....

b) Determine the concentration of iodine in FA2 in gdm^{-3} :

Standardization of sodium thiosulphate solution using potassium iodine

You are provided with the following;

FA1, which is a solution made by dissolving 1.07g of potassium iodate to make a 250cm³solution.

FA2, which is a solution containing sodium thiosulphate, $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$.

0.5M potassium iodide solution.

2M sulphuric acid

Procedure:

Pipette 25.0 (or 20.0) cm³ of FA2 into a conical flask, add 10cm³ of 0.5M KI followed by 10cm³ of 2M sulphuric acid. Titrate the resultant mixture with FA2 from the burette until the solution is pale yellow. Add 5 drops of starch indicator and continue with the titration until endpoint.

Repeat the procedure to obtain consistent titre values and record your results in the table below.

Results

Volume of pipette used: cm³

Burette readings:

Final reading (cm ³)			
Initial reading (cm ³)			
Volume of FA2 used (cm ³)			

Average volume of FA2 used

Write equation(s) that occurred

Questions.

a) Calculate:

i) Moles of potassium iodate (FA1) that reacted:

ii) Moles of thiosulphate that reacted:

b) concentration of sodium thiosulphate in grams per litre

.....

 Determination of percentage of potassium iodate in an impure sample

You are provided with the following;

FA1, which is made by dissolving 8.90g of a mixture potassium iodate with potassium iodide to make 1 litre of solution.

FA2, which is a solution made by dissolving 12.4g of sodium thiosulphate-5-water in 250cm³ of solution.

0.5M potassium iodide solution.

2M sulphuric acid

(K =39, I =127, O =16)

Procedure:

Pipette 25.0 or 20.0 cm³ of FA1 into a conical flask, add 20cm³ of 0.5M KI followed by 20cm³ of 2M sulphuric acid. Titrate the resultant mixture with FA2 from the burette until the solution is pale yellow. Add 5 drops of starch indicator and continue with the titration until endpoint.

Repeat the procedure to obtain consistent titre values and record your results in the table below.

Results

Volume of pipette used: cm³

Burette readings:

Final reading (cm ³)			
Initial reading (cm ³)			
Volume of FA2 used (cm ³)			

Average volume of FA2 used

.....

Questions.

a) Calculate:

i) Molar concentration of KIO₃ in FA1

.....

ii) Mass of KIO₃ in 1 litre of FA1

.....

b) determine the percentage of KIO₃ in the mixture.

Standardising Sodium thiosulphate and determination of chlorine in bleaching agent

You are provided with the following:

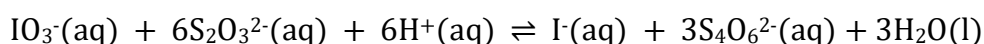
FA1; which is 0.012M potassium iodate solution.

W, which is sodium thiosulphate, $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$.

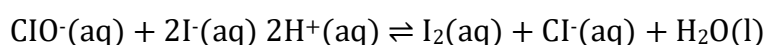
Z, which is aliquid bleaching agent containing chlorate(I).

You are required to prepare and standardize a solution of sodium thiosulphate and use it to determine the mass of chlorine required to produce 1 dm³ of the bleaching agent.

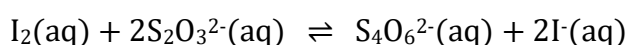
Potassium iodate reacts with sodium thiosulphate according to the following equation;



Chlorate(1) reacts with potassium iodide to form iodine according to the following equation;



And iodine reacts with sodium thiosulphate according to the following equation;



Procedure

- (a) Weigh about 6.0g of W and dissolve it in about 100cm³ of distilled water . transfer the solution to a 250cm³ volumetric flask and make it up to the mark with distilled water. Label the solution FA2.
Pipette 25.0 (or 20.0) cm³ of FA1 into a conical flask. Add 20cm³ of 1M sulphuric acid, followed by 20cm³ of 0.5M potassium iodide solution. Titrate the mixture with FA2 until the brown colour changes yellow. Add 1cm³ of starch indicator and continue the titration until the solution turns colourless.
Repeat the titration until you obtain consistent results.

Record your results in table A

Results

Mass of weighing vessel + N =g

Mass of weighing vessel alone =g

Mass of solid N alone =.....g

Volume of pipette used:.....cm³

Table A

Final reading (cm ³)			
Initial reading (cm ³)			

Volume of FA2 used (cm ³)			
---------------------------------------	--	--	--

Volume of FA2 used in calculating average volume

.....cm³

Average volume of FA2 used.....cm³

Question:

(i) Calculate the molarity of FA2.

.....

.....

.....

.....

.....

.....

(b) Measure 10cm³ of Z into a 250cm³ volumetric flask. Add distilled water up to the mark. Label the solution FA3.

Pipette 25.0 or 20.0cm³ of FA3 into a conical flask. Add 10cm³ of 0.5M potassium iodide followed by 10cm³ of 1M sulphuric acid. Titrate the resultant mixture with FA2 until the solution turns yellow. Add 1cm³ of starch indicator and continue the titration until the solution turns colourless.

Repeat the procedure to obtain consistent titre values.

Record your results in table B.

Results

Volume of pipette used =.....cm³

Final reading (cm ³)			
Initial reading (cm ³)			
Volume of FA2 used (cm ³)			

Volume of FA2 used in calculating average volume

.....cm³

Average volume of FA2 usedcm³

Question:

(i) The moles of iodine liberated.

.....

.....

.....

.....

.....

(ii) The moles of chloride ions that would be liberated from 10cm³ of Z.

.....

.....

.....

.....

(iii) The moles of chlorine required to produce 1dm³ of Z

.....

.....

.....

(iv) Determine the mass of chlorine require to produce 1 dm³ of Z.[Cl=35.5]

.....

.....

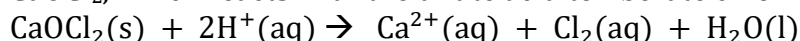
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Estimation of available chlorine' in a bleaching powder

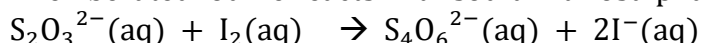
You are provided with the following
 FA1, which is 0.1M sodium thiosulphate
 10% potassium iodide
 Bleaching powder
 Ethanoic acid
 Starch solution

Theory

'Available chlorine refers to chlorine liberated when bleaching powder reacts with a dilute acid e.g. HCl. The active ingredient in a bleaching powder is calcium hypochlorite, CaOCl₂, which reacts with the dilute acid to liberate chlorine.



The liberated iodine reacts with sodium thiosulphate when titrated as;



Procedure:

Weigh accurately 2.5g of bleaching powder into a clean mortar. Add about 50cm³ of water and grind the mixture into a fine paste.

Transfer the paste into a 250cm³ volumetric flask and wash off the remains into the flask with water. Make the solution to the mark with more water and shake well. Label the solution FA2

Pipette 50cm³ of FA2 into a clean conical flask. Add 20cm³ of 10% potassium iodide and 10cm³ of ethanoic acid. Titrate the mixture with FA1 until it turns pale yellow. Then add starch indicator and continue the titration until the solution becomes colourless.

Repeat the procedure to obtain consistent titre values and record your results in the space below.

Results:

Mass of weighing vessel t bleaching powder =g

Mass of weighing vessel alone =g

Mass of bleaching alone =g

Volume of pipette used = cm³

Burette readings

Final burette reading/cm ³			
Initial burette reading/cm ³			
Volume of FA1 used /cm ³			

Titre values used to calculate the average volume of FA1 used

.....

Average volume of FA31 used

.....

a) Calculate

i) moles of thiosulphate that reacted.

.....

ii) moles of iodine that was liberated

.....

iii) moles of chlorine liberated

.....

iv) the percentage of chlorine in the bleaching powder.

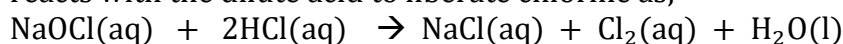
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Estimation of 'available chlorine' in JIK

You are provided with the following
 FA1, which is 0.1M sodium thiosulphate
 10% potassium iodide
 Starch solution

Theory

JIK is a liquid that bleaches. Its activity ingredient is sodium hypochlorite (NaOCl) which reacts with the dilute acid to liberate chlorine as;



The liberated chlorine reacts with potassium iodide to produce iodine that can be titrated against sodium thiosulphate

Procedure

Accurately measure 10cm³ of JIK and dilute it to 250cm³ with distilled water in a volumetric flask. Label the solution FA2.

Pipette 25.0 or 20.0cm³ of FA2 into a clean conical flask. Add 15cm³ of sulphuric acid and 15cm³ of 10% potassium iodide. Titrate the mixture with FA1

Repeat the procedure to obtain consistent titre values.

Record your results in the space below.

Results:

Volume of pipette used =cm³

Burette readings

Final burette reading/cm ³			
Initial burette reading/cm ³			
Volume of FA1 used /cm ³			

Titre values used to calculate the average volume of FA1 used

.....

Average volume of FA1 used

.....

a) Calculate

i) moles of thiosulphate that reacted.

.....

ii) moles of iodine that was liberated

.....

iii) the percentage of chlorine in the JIK

.....

THERMOCHEMISTRY

This is the study of heat changes that accompany chemical reactions.

Enthalpy is the heat content of a substance.

Enthalpy change is the heat change that occurs during a chemical reaction. Its either positive or negative and is always written at the end of the chemical equation.

E.g. $C(s) + O_2(g) \rightarrow CO_2(g)$; $\Delta H = -393 \text{ KJ/mol}$

Exothermic and Endothermic reactions

Exothermic Reactions

They are reactions that release heat energy to the surrounding. Release of heat is detected by increase in temperature of the surroundings. Enthalpy change (ΔH) for an exothermic reaction is negative.

Endothermic Reactions

Are reactions that absorb heat energy from the surrounding. Heat absorption is detected by decrease in temperature. Enthalpy change (ΔH) for an endothermic reaction is positive.

Determination of enthalpy of solution of sodium hydroxide

You are provided with the following:

4g of sodium hydroxide

Measuring cylinder

Plastic cup or beaker

Thermometer

Distilled water

Procedure

- Measuring 90.0cm³ of water into a plastic cup and record its initial temperature t_1
- Transfer the 4g of sodium hydroxide into water. Stir carefully using the thermometer and record the final temperature t_2

Results

$t_1 =$

$t_2 =$

a) Calculate

i) The change in temperature $\Delta t =$

ii) The mass of solution takes the density of solution = 1gmcm⁻³)

iii) The molar heat of solution of sodium hydroxide (assume that the specific heat of the solution = 4.3 J mol⁻¹ °C⁻¹; Na =23, O = 16, H=1)

.....

.....

.....

.....

.....

.....

.....

.....

b) State whether the reaction is exothermic or endothermic. Explain

.....

.....

.....

Determination of enthalpy of neutralization of hydrochloric acid by sodium hydroxide solution

You are provided by the following

YA1; which is 2M hydrochloric acid

YA2; which is 2M sodium hydroxide solution

Measuring cylinder

Plastic cup or beaker

Thermometer

Procedure

- Measure 50cm³ of YA1 into a plastic beaker. Stir carefully using the thermometer and record the initial temperature t_1 , of YA1
- measure 50cm³ of YA2 into a plastic beaker. Stir carefully using the thermometer and record the initial temperature t_2 , of FA2
- Immediately mix the solutions while stirring with a thermometer. Record the highest temperature t_3 attained by the solution.

Results

Initial temperature of YA1 =.....

Initial temperature of YA2 =.....

Average initial temperature of YA1 and YA2

.....

Find temperature of solution =

Change in temperature Δt =

Questions:

- Write equation for the reaction that takes place

.....

- Calculate the heat change for the reaction (take the density of solution = 1gcm⁻³, assume that the specific heat of the solution = 4.2 Jmol⁻¹°C⁻¹)

.....

.....

.....

.....

.....

- Calculate the moles of FA1 and FA2 that reacted

.....

.....

.....

.....

.....

- Hence calculate the molar heat of neutralization of FA1 by FA2.

.....

...

.....

- Is the value in b (iii) positive or negative? Explain

.....

.....

.....

Determination of enthalpy of neutralization of hydrochloric acid by sodium hydroxide using thermometric titration

You are provided with the following acid
FA1; which is 2M hydrochloric acid

FA2; which is 2M sodium hydroxide solution

Measuring cylinder

Plastic cup or beaker

Thermometer

Burette

Procedure:

- Record the initial temperature of FA1, then
- Fill the burette with FA1
- Measure 50cm³ of FA2 into a plastic beaker and record its initial temperature t_1
- Add FA1 from the burette to FA2 in 5.0cm³ portions at regular time intervals. Carefully stir the mixture using a thermometer. Note and record the temperature.
- Repeat procedure c) using 10cm³, 15cm³, 20cm³, 25cm³, 30cm³, 35cm³, 40cm³, 45cm³ and 50cm³. Record your results in the table below.

Results:

Initial temperature of FA1 =

Initial temperature of FA2 =

Average initial temperature of FA1 and FA2 =

Table of results

Volume of BA1 used/cm ³	0	5	10	15	20	25	30	35	40	45	50
Temperature/ °C											

- Plot a graph of temperature (°C) against volume of acid (FA1) added (cm³)

From the graph determine the;

- Volume of acid required to neutralize FA2

.....

- Maximum temperature rise

.....

- Calculate the enthalpy change for the reaction (take the density of solution = 1gcm⁻³, assume that the specific heat of the solution = 4.2 J mol⁻¹°C⁻¹)

.....

- State and explain the nature of the graph obtained if 2M ethanoic acid is used instead of 2M hydrochloric acid.

.....

Determination of enthalpy of displacement of Cu²⁺ by zinc powder or iron fillings

You are provided with the following

0.2M solution of copper (ii) sulphate

Iron fillings

Measuring cylinder

Plastic cup
Thermometer

Procedure:

- Measure 50cm³ of 0.2M copper(ii) sulphate solution into a plastic beaker and record its initial temperature t_1
- Transfer all the iron fillings provided into the cup containing the copper (ii) sulphate solution. Stir the mixture carefully using a thermometer and record the highest temperature reached, t_2

Results:

Initial temperature of the solution =

Initial temperature of the solution =

Change in temperature Δt =

Questions

- Write an ionic equation for the reaction that takes place

.....
.....

- Calculate the heat change for the reaction

.....
.....
.....
.....
.....

- Hence determine the enthalpy of displacement of Cu²⁺ by iron fillings

.....
.....
.....
.....

- Why is it necessary to use excess iron fillings in this experiment?

.....
.....

CHEMICAL KINETICS

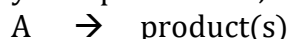
Kinetics is the study of speeds at which reactions occur and factors which influence the reaction. These factors include; temperature, concentration, surface area of reactants, Pressure and catalysts.

Terms used in kinetics

Order of reaction: is the power to which the concentration term of a reactant is raised in a rate equation.

Rate equation is a mathematical relationship that relates the rate of reaction to the concentration of a reactant to an appropriate power.

If A is a reactant that yields products as;



The rate equation is

$$\text{Rate} \propto [A]^n$$

$$\text{Rate} = K[A]^n$$

Where K = rate constant

n = order of reaction

How to use shapes to deduce order of reaction depending on the axes of the graph. the axes are derived from the integrated rate equation for the particular order of reaction.

Iodination of propanone

You are provided with the following

FA1; 0.05M iodine solution

FA2; 1.0M propanone

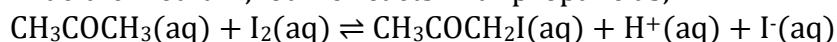
FA3; 0.1M sodium thiosulphate

FA4; 0.5M sodium hydrogen sulphate

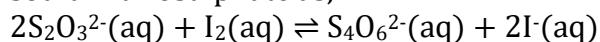
1.0M sulphuric acid

Theory

In acidic medium, iodine reacts with propane as;



The amount of unreacted iodine is determined by titration with standard solution of sodium thiosulphate as;



The volume of the thiosulphate required to reach the end point is proportional to the volume of the un decomposed iodine.

Procedure

- Pipette 25cm³ of FA2 and 25cm³ of 1.0M sulphuric acid into a conical flask.
- Add 50cm³ of FA1 all at once and start the stop clock simultaneously. Shake the mixture well and keep it for subsequent procedures
- Label 5 other conical flasks as 1,2,3,4 and 5
- Transfer 10cm³ of into each conical flask
- Add 10cm³ of the mixture in (b) to flask 1 at exactly 5 minutes from the start of the stop clock. Shake well and titrate the residual iodine with FA3 from the burette using starch indicator. Record your results in the table below.
- Repeat the procedure (e) with flasks 2, 3,4 and 5 at 10, 15, 20 and 25 minutes respectively.

Results

Flask	1	2	3	4	5
Time/Min	5	10	15	20	25
Final burette reading /cm ³					
Initial burette reading /cm ³					
Volume of Fa3 used / cm ³					

Questions

- Plot a graph of volume of thiosulphate against time in minutes
- Use the graph to deduce the order of reaction with respect to iodine. Give a reason for your answer.
- Use the graph to predict the volume of sodium thiosulphate which reacts with 10cm³ of the reaction mixture at t=0 minutes
- Explain why sodium hydrogen sulphate was added to the mixture before titration.

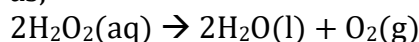
Determination of order of reaction with respect to hydrogen peroxide

You are provided with the following

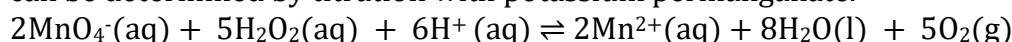
FA1; Hydrogen peroxide
 FA2; 0.02M potassium permanganate
 FA3; 0.5M iron (iii) chloride
 FA4; 2M sulphuric acid
 1.0M sodium hydroxide
 Stop clock

Theory

Catalysed by iron (iii) chloride, hydrogen peroxide decomposes under alkaline conditions as;



Adding an acid stops the decomposition. Hence the undecomposed hydrogen peroxide can be determined by titration with potassium permanganate.



The volume of permanganate used is proportional to the volume of hydrogen peroxide undecomposed.

Procedure

- Pipette 10cm³ of FA1 into a conical flask; add 2cm³ of 1M sodium hydroxide followed by 5cm³ of FA3. Start the stop clock immediately.
- Shake the flask and leave it stand for 2 minutes and 100cm³ of FA4. Titrate the mixture with FA4 until it turns pink. Record your results in the table below.
- Repeat procedures (a) and (b), allowing the flask stand for 4, 6, 8, 10, 12 and 14 minutes

Results

Time / Mins	2	4	6	8	10	12	14	16
Volume (V _t) of FA2 /cm ³								
Log ₁₀ of V _t								

Questions

- Plot a graph of Log₁₀V_t against time in minutes
- Use the graph to deduce the order of reaction with respect to hydrogen peroxide. Give a reason

.....

.....

- Determine the slope of your graph and hence find the value of the rate constant.

.....

.....

.....

.....

COLLIGATIVE PROPERTIES

Determination of freezing point and freezing point depression constant K_f.

F is camphor

G is naphthalene

You are provided with solids F and G

You are required to determine the depression point of F, and the freezing point depression constant, K_f per kg of F.

Procedure:

- (a) Pour about 200cm³ of water into a 250cm³ beaker. Heat the water to boiling on a tripod stand.
- (b) In the meantime, weigh separately and accurately about 5.0g of F and 1.0g of G. Record the results of your measurements in the space provided below.

Results:

Mass of container + F =

Mass of empty container =

Mass of F =

Mass of container + G =

Mass of empty container =

Mass of G =

- (c) Transfer the whole of F into a clean dry boiling tube. Immerse the boiling tube containing F into the beaker of hot water and continue heating the water until the whole of F melts.
- (d) Insert a thermometer in the liquid formed and heat to about 87°C. Remove the boiling tube from the hot water and start the stop clock when its temperature drops to 85°C. Allow the liquid to cool while stirring with the thermometer and record its temperature after every half a minute for three minutes. Enter your results in the table below.
- (e) Transfer the whole of G into a boiling tube containing F. Immerse the boiling tube into the beaker of hot water and continue heating the water until the mixture melts. Continue heating until the temperature of the molten mixture is about 87°C. While stirring gently with thermometer, allow the mixture to cool and record its temperature after every half a minute for three minutes.

- (f) Enter your results in the table below

Time (Seconds)	0	30	60	90	120	150	180
Temperature of pure F (°C)							
Temperature of mixture of F and G (°C)							

Questions

- (a) Plot on the same axes, a graph of temperature of

- (i) Pure F against time
(ii) Mixture of F and G against time.

(use graph paper on the next page)

- (b) Describe the shapes of the graphs you have drawn.

.....
.....
.....

- (c) From your graphs, read off the temperature after 2.5 minutes of

- (i) Pure F
(ii) Mixture of F and G

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 (d) Use the temperatures you have obtained in (c) above to determine the depression in freezing point of F

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 (e) Calculate the freezing point depression constant, K_f per Kg of F. (RFM of solid G is 152)

QUALITATIVE ANALYSIS

This involves two parts:

1. Organic qualitative analysis – which deals with identifying cations and anions present in a given sample.
2. Organic qualitative analysis – which deals with identifying the nature of a given sample of an organic compound.

QUALITATIVE INORGANIC ANALYSIS

Preliminary inorganic qualitative analysis

Anions are predicted in association with the gases evolved during some tests and some are confirmed by reactions involving precipitation.

Cations are commonly predicted from colours of solids, solutions and reaction with certain reagents

Effect of heat

When heated, some samples decompose to give off gases and a residue remains in the test tube. The gases are identified by their colour, effect on litmus paper and confirmatory test. The sample is heated in a dry test tube until there is no further change.

Examples:

Observation	Deduction (conclusion/inference)
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Colourless gas turns litmus to red and lime water milky.	CO ₂ gas evolved
Colourless gas turns red litmus blue, forms dense white fumes with hydrogen chloride gas.	NH ₃ gas evolved. NH ₄ ⁺
Misty fumes turns blue litmus red, from dense white fumes with NH ₃ (g)	HCl(g) Cl ⁻
White fumes, turns blue litmus red and acidified Ba(NO ₃) ₂ milky	SO ₃ (g), SO ₄ ²⁻
Colourless gas turns blue litmus to red, turns acidified potassium dichromate from orange to green (or acidified potassium manganate (VII))	SO ₂ (g) SO ₄ ²⁻ , SO ₃ ²⁻ suspected
Brown gas turns blue litmus red, and a colourless gas that relights a glowing splint	NO ₂ (g) therefore NO ₃ ⁻ O ₂ (g) evolved
Colourless vapour condenses to colourless liquid, turns anhy. CuSO ₄ to blue	Water vapour, therefore hydrated salt (water of crystallization)
Residue is yellow when hot, white when cold	ZnO
Residue is reddish brown when hot, yellow when cold	PbO
Residue black	CuO, NiO, FeO
Residue is brown	Fe ₂ O ₃
Residue is white	Al ₂ O ₃ , MgO, CaO

Identification of Cations

1. Colour of solids:

- White solids contain compounds of Lead, Aluminium, Zinc, Magnesium, Calcium, Ammonia, Tin or Barium ions.
- Pink solid contains Manganese or cobalt
- Blue solids contain compounds of copper or cobalt.
- Green solids contain compounds of Cu²⁺, Fe²⁺, Ni²⁺, Cr³⁺
- Brown or yellow solids are compounds of Iron(III). FeCl₃ is black

2. Colour of solutions:

- Colourless solutions contain cations of metals that form white solids e.g. Pb²⁺, Al³⁺, Ca²⁺, NH₄⁺, Sn²⁺, Ba²⁺
- Blue solutions contain Cu²⁺ or Ni (NH₃)₆²⁺
- Green solutions contain Fe²⁺, Cu²⁺, Ni²⁺ or Cr³⁺
- Yellow solutions contain Fe³⁺ or Cr₂O₇²⁻
- Purple solutions contain MnO₄⁻
- Pink solutions contain Mn²⁺ or Co²⁺.

Effects of sodium hydroxide solution.

Sodium hydroxide solution contains hydroxide ions (OH^-) which combine with cations to form hydroxides. Since most hydroxides are insoluble in water, precipitates are formed.

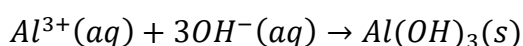
Procedure: add dilute sodium hydroxide solution drop-wise until in excess.

Observation	Deduction
White precipitate, dissolves, forming a colourless solution	Zn^{2+} , Al^{3+} , Pb^{2+} , Sn^{2+}
White precipitate, insoluble in excess, NaOH	Mg^{2+} , Ca^{2+} , Ba^{2+}
White precipitate, insoluble in excess, rapidly turns brown	Mn^{2+}
Blue precipitate, insoluble in excess, turns pink on standing	Co^{2+}
Blue precipitate, insoluble in excess NaOH	Cu^{2+}
Green precipitate soluble in excess NaOH forming a green solution	Cr^{3+}
Green precipitate soluble in excess NaOH (in case of iron, precipitate turns brown on standing)	Fe^{2+} , Ni^{2+}
Brown precipitate, insoluble in excess NaOH	Fe^{3+}
(if warming is done) colourless gas, turn red litmus to blue.	$\text{NH}_3(\text{g})$ evolved NH_4^+ confirmed

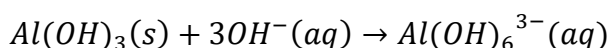
Explanation

Precipitates are due to formation of insoluble hydroxides e.g. $\text{Al}(\text{OH})_3$, $\text{Zn}(\text{OH})_2$, $\text{Cr}(\text{OH})_3$

Sample equation:



Some precipitates dissolve in excess NaOH because their hydroxides are amphoteric and hence react with the excess NaOH to form soluble salt complexes. E.g. Aluminium hydroxide, zinc hydroxide and lead(II) hydroxide dissolve



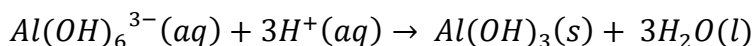
$\text{Mg}(\text{OH})_2$ and $\text{Ca}(\text{OH})_2$ are not amphoteric and hence insoluble in excess NaOH.

Task: Explain each of the observations made in the table above. Write equations in each case.

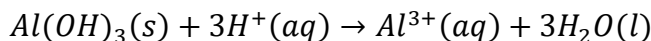
NB: Consider a white precipitate that dissolves in excess sodium hydroxide.

When dilute Hydrochloric acid or nitric acid is added drop-wise, the precipitate reappears due to formation of the insoluble hydroxide and further addition of the acid dissolves the precipitate by neutralization.

Example: Addition of dilute hydrochloric acid to the colourless solution of the salt complex formed when excess sodium hydroxide is added a solution containing aluminium ions. A white precipitate of aluminium hydroxide is formed.

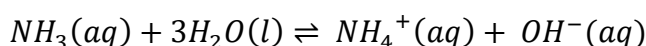


Further addition of acid dissolves the white precipitate to form a colourless solution.



Effects of ammonia solution.

Ammonia solution is a weak alkali solution made by dissolving ammonia in water. Ammonia solution therefore contains hydroxide ions (OH^-).



These hydroxide ions combine with metal cations to form metallic hydroxides. Since most hydroxides are insoluble in water, precipitates are formed. Therefore, most reaction of ammonia are similar to those of sodium hydroxide.

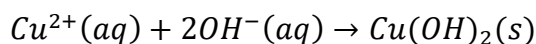
It is important to note that ammonia is weak alkali and contains ammonia (NH_3) molecules.

Procedure: add dilute ammonia solution drop-wise until in excess.

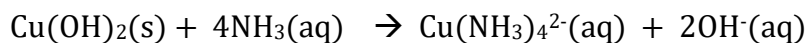
Observation	Deduction
White precipitate, dissolves in excess ammonia solution, forming a colourless solution	Zn^{2+}
White precipitate, insoluble in excess ammonia solution	$Al^{3+}, Pb^{2+}, Sn^{2+}$
White precipitate, insoluble in excess, rapidly turns brown	Mn^{2+}
White precipitate, insoluble in excess, ammonia solution	Mg^{2+}, Ba^{2+}
Blue precipitate, dissolves in excess, turns red on standing	Co^{2+}
Blue precipitate, dissolves in excess ammonia solution forming a deep blue solution	Cu^{2+}
Green precipitate, dissolves to form a violet solution	Cr^{3+}
Green precipitate, dissolves to form a blue solution	Ni^{2+}
Green precipitate, insoluble in excess ammonia solution.	Fe^{2+}
Brown precipitate, insoluble in excess ammonia solution	Fe^{3+}

Explanation

Precipitates are due to formation of an insoluble hydroxides e.g $Cu(OH)_2$.

Equation:


However, $\text{Zn}(\text{OH})_2$ and $\text{Cu}(\text{OH})_2$ dissolve in excess ammonia solution due to formation of soluble complexes. E.g.



Task: Explain each of the observations made in the table above. Write equations in each case

Confirmatory Tests

Non-transition metal cations:

Lead (ii) ions, Pb^{2+} .

i) Add 2-3 drops of potassium iodide solution.

Observation	Deduction
Yellow precipitate	Pb^{2+} confirmed

ii) Add 2-3 drops of HCl solution (Cl^{-} ions), warm and allow cooling.

Observation	Deduction
White precipitate, dissolves on warming, reappears on cooling	Pb^{2+}

iii) Add 2 to 3 drops of potassium chromate(VI) followed by 2 to 3 drops of sodium hydroxide solution.

Observation	Deduction
Yellow precipitate, dissolves sodium hydroxide to form a yellow solution.	Pb^{2+}

Zinc ions, Zn^{2+}

Add a spatula of solid ammonia chloride followed by 3 to 4 drops disodium hydrogen phosphate solution and then aqueous ammonia dropwise until in excess.

Observation	Deduction
White precipitate, dissolves in excess ammonia	Zn^{2+}

Aluminium ions, Al^{3+}

Add 2 to 3 drops of litmus solution, followed by NH_3 solution dropwise until in excess.

Observation	Deduction
A blue 'lake' formed	Al^{3+}

Barium ions, Ba^{2+}

i) Add potassium chromate solution followed by ethanoic acid solution.

Observation	Deduction
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Yellow precipitate, insoluble in acid	Ba ²⁺ confirmed
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ii) Add 2 to 3 drops of K₂CrO₄ followed by 2 to 3 drops of NaOH solution.

Observation	Deduction
Yellow precipitate, insoluble in sodium hydroxide.	Ba ²⁺

Magnesium ions, Mg²⁺

Add a spatula of solid NH₄Cl followed by 3 to 4 drops disodium hydrogen phosphate solution and then aqueous NH₃ drop wise until in excess

Observation	Deduction
White precipitate, insoluble in excess ammonia	Mg ²⁺

Calcium ions Ca²⁺

Add potassium chromate solution followed by ethanoic acid solution.

Observation	Deduction
Yellow precipitate, dissolves in acid	Ca ²⁺ confirmed

Transition metal cations

Iron(II) ions, Fe²⁺

Add potassium hexacyanoferrate(III) solution.

Observation	Deduction
Blue precipitate	Fe ²⁺ confirmed

Iron(III) ions, Fe³⁺.

(i) Add potassium hexacyanoferrate(II) solution.

Observation	Deduction
Blue precipitate	Fe ³⁺ confirmed

(ii) Add potassium/ammonium thiocyanate.

Observation	Deduction
Red solution	Fe ³⁺ confirmed

Copper(II) ions, Cu²⁺

(i) Add potassium hexacyanoferrate(II) solution.

Observation	Deduction
Brown precipitate	Cu ₂ Fe(CN) ₆ formed; Cu ²⁺ confirmed

(ii) Add 2-3 drops of potassium iodide solution

Observation	Deduction
White precipitate in a brown solution	Cu ₂ I ₂ formed; Cu ²⁺ confirmed

Manganese(II) ions, Mn^{2+}

Add conc. HNO_3 followed by sodium bismuthate solid and heat

Observation	Deduction
Purple solution formed	MnO_4^- formed; Mn^{2+} confirmed

Note. Lead(IV) oxide can be used in place of sodium bismuthate($NaBiO_3$).

Chromium(III) ions, Cr^{3+}

Add excess sodium hydroxide followed by hydrogen peroxide and heat.

Observation	Deduction
Yellow solution	CrO_4^{2-} formed; Cr^{3+} confirmed

Nickel(II) ions

Add ammonia solution followed by 2 drops of dimethylglyoxime.

Observation	Deduction
Red precipitate	Ni^{2+} confirmed

Cobalt(II) ions, Co^{2+}

Add potassium thiocyanate solution.

Observation	Deduction
Blue solution	Co^{2+}

Identification of Anions

Effect of acids on solids or solution

Procedure, Observation and Deductions:

(i) Add dilute HCl (or HNO_3) to the solid

Observation	Deduction
Solid dissolves with effervescence of a colourless gas that turns limewater milky	$CO_2(g)$ evolved Therefore CO_3^{2-} present

(ii) Add dilute HCl (or HNO_3) to the solution.

Observation	Deduction
Colourless gas, turns acidified potassium dichromate from orange to green	$SO_2(g)$ evolved SO_3^{2-} present

(iii) Add conc. H_2SO_4 to the solid.

Observation	Deduction
Misty fumes, form dense white fumes with ammonia	HCl(g) evolved; Cl ⁻ present

Effect of Lead(II) nitrate solution.

(i) Add 2-3 drops of lead(II) nitrate solution

Observation	Deduction
White precipitate,	Cl ⁻ , SO ₄ ²⁻
Yellow precipitate	I ⁻
White precipitate dissolves on warming, reappears on cooling	Cl ⁻
White precipitate persists on warming.	SO ₄ ²⁻

(ii) Add 2-3 drops of lead(II) nitrate solution followed by dilute nitric acid solution.

Observation	Deduction
White precipitate, insoluble in acid	Cl ⁻ , SO ₄ ²⁻ present
White precipitate, gas that turns acidified K ₂ Cr ₂ O ₇ green.	SO ₃ ²⁻

Confirmatory Tests for Anions

Sulphate ions (SO₄²⁻)

(i) Add dilute HNO₃ followed by Barium nitrate solution.

Observation	Deduction
White precipitate	SO ₄ ²⁻ ion confirmed

(ii) Add barium nitrate solution followed by dilute HNO₃.

Observation	Deduction
White precipitate insoluble in acid	SO ₄ ²⁻ ions confirmed

Note: Barium chloride can be used with hydrochloric acid.

Sulphate ion (SO₃²⁻)

(i) Add acidified potassium permanganate solution

Observation	Deduction
Purple solution of permanganate turns colourless	SO ₃ ²⁻ present

(ii) Add acidified potassium dichromate solution.

Observation	Deduction
Orange colour of dichromate turned green	SO_3^{2-} present

Chloride ions (Cl^-).

Add dilute nitric acid followed by silver nitrate solution

Observation	Deduction
White precipitate	Cl^- ions

Nitrate ion (NO_3^-).

Add equal volume of freshly prepared Iron(II) sulphate followed by conc. H_2SO_4 added drop-wise down the sides of a slanting test tube.

Observation	Deduction
Brown ring is formed	NO_3^- ions confirmed

Carbonate (CO_3^{2-})

(i) Add dilute HCl (or HNO_3) to the solid.

Observation	Deduction
Solid dissolves with effervescence of a colourless gas that turns lime water milky	$\text{CO}_2(\text{g})$ evolved Therefore CO_3^{2-} present

(ii) Add magnesium sulphate to solution.

Observation	Deduction
White precipitate	CO_3^{2+}

Ethanoate/acetate ion (CH_3COO^-)

(i) Add about 2cm^3 of ethanol followed by 2-3 drops of conc. H_2SO_4 and warm

Observation	Deduction
Fruity smelling liquid	CH_3COO^-

(ii) Add about 2-3 drops of neutral iron(III) chlorine solution.

Observation	Deduction
Reddish brown colouration	CH_3COO^- confirmed

Oxalate ion ($\text{C}_2\text{O}_4^{2-}$)

Add acidified potassium permanganate and heat

Observation	Deduction
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Permanganate solution turns colourless, a colourless gas turns lime water milky	$\text{C}_2\text{O}_4^{2-}$ confirmed
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Inorganic qualitative analysis Practical Tasks

Task 1

You are provided with substance E, which contains one cation and one anion. carry out the following tests on E and identify any gas(es) evolved. Record your observations and deductions in the spaces provided.

Test	Observation	Deduction
a) Heat two spatula endful of E strongly in a dry test tube.		
b) To one spatula endful of E add dilute HNO_3 until there is no further change. Divide the resultant solution into 3 portions.		
i) To the first portion of the solution add dilute NaOH drop-wise until in excess.		
ii) To the second portion of the solution add dilute ammonia solution drop-wise until in excess		
iii) To the third portion, carry out a test of your own to confirm the cation in E		

c) Identify

- The cation in E:
- The anion in E:

Task 2

You are provided with substance F, which contains two cation and two anions. carry out the following tests on F and identify any gas(es) evolved. Record your observations and deductions in the spaces provided.

Test	Observation	Deduction
a) Heat two spatula endful of F strongly in a dry test tube.		

b) Dissolve two spatula endful of F in about 8cm ³ of distilled water. Filter, keep both residue and filtrate. Divide the filtrate into five portions.		
i) To the first portion of the filtrate add NaOH drop-wise until in excess.		
ii) To the second portion of the filtrate add dilute NH ₃ solution drop-wise until in excess		
iii) Use 3 rd portion to carryout own test to confirm cation		
iv) To the 4 th portion of the filtrate add 3 drops of Pb(NO ₃) ₂		
v) To the 5 th portion carry out a test of your own to confirm the anion present		
c) Wash the residue and transfer it to a clean test tube. Add distilled HNO ₃ until all the residue dissolves. Divide the resultant solution into 3 portions		
i) To the first portion of solution add sodium hydroxide drop-wise until in excess.		
ii) To the second portion of solution add ammonia solution drop-wise until in excess.		
iii) To the third portion, carry out a test of your own to confirm the cation in F		

- b) Name:
- i) the cations in F
- ii) the anions in F

Task 3

You are provided with substance W, which contains one cation and one anion. carry out the following tests on W and identify any gas(es) evolved. Record your observations and deductions in the spaces provided.

Test	Observation	Deduction
a) Heat two spatula endful of W strongly in a dry test tube.		
b) Dissolve two spatula end-full of W in about 5cm ³ of water and divide the solution into five portions		
i)to the first portion of the solution add dilute NaOH drop-wise until in excess.		
ii)to the second portion of the solution add dilute ammonia solution drop-wise until in excess		
iii)to the third portion, of the solution add 2-3 drops of potassium hexacyanoferrate(iii).		
iv)to the fourth portion add 2-3 drops of lead(II) nitrate solution		
v)to the fifth portion add excess dilute nitric acid followed by 2-3 drops of barium nitrate solution		

c) Identify

- i) the cation in W.....
- ii) the anion in W.....

Task 4

You are provided with substance G, which contains two cation and two anions. Carry out the following tests on G and identify any gas (es) evolved. Record your observations and deductions in the spaces provided.

Test	Observation	Deduction
a) Heat two spatula endful of F strongly in a dry test tube.		
(b) Dissolve two spatula endful of G in about 5cm ³ of		

water. Divide the solution into six portions.		
i) To the first portion add NaOH drop-wise until in excess.		
ii) To the second add ammonia solution drop-wise until in excess		
(iii) To the third portion of the solution add a spatula endful of iron filings, shake and allow to stand.		
(iv) Use the 4 th portion to carry out a test of your own to confirm the cation present		
(v) To the 5 th portion add 2-3 drops of $\text{Pb}(\text{NO}_3)_2$ followed by dilute HNO_3 and boil.		
(vi) To the 6 th portion carry out test of your own to confirm the anion in G.		

- c) Identify:
- the cations in W
 - the anions in W
- c) Explain your observation in test (b)(ii).

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Task 5

You are provided with substance M, which contains two cation and one anion. carry out the following tests on M and identify any gas(es) evolved. Record your observations and deductions in the spaces provided.

Test	Observation	Deduction
a) Heat two spatula endful of M strongly in a dry test tube.		
b) Add dilute HNO_3 to M until no further change. To the resultant		

	solution add NaOH solution until no further change and filter. Keep both filtrate and residue		
c)	To filtrate add dilute HNO_3 drop-wise until no further change. Divide the resultant solution into three parts.		
i)	To the first portion of the solution add dilute sodium hydroxide solution drop-wise until in excess		
ii)	To the second portion of the solution add dilute ammonia solution drop-wise until in excess		
iii)	To the third portion add 2-3 drops of potassium iodide solution		
d)	Wash the residue with sodium hydroxide followed by water then dissolves in dilute hydrochloric acid and divides the resultant solution into three parts.		
i)	To the first portion of solution add dilute NaOH solution drop-wise until in excess.		
ii)	To the second portion add dilute $\text{NH}_3(\text{aq})$ solution drop-wise until in excess		
iii)	To 3 rd portion add 2-3 drops of potassium iodide solution.		

d) Identify:

i) the cations in M

ii) the anions in M

Task 6

You are provided with substance D, which contains two cation and two anions. carry out the following tests on D and identify the cations and anions in D. Identify any gas(es) evolved. Record your observations and deductions in the spaces provided.

Test	Observation	Deduction
a) Heat two spatula endful of D strongly until there is no further change.		
b) Dissolve two spatula end-full of D, in about 8cm ³ of water, filter, and keep both residue and filtrate. divide the filtrate into four portions		
i)to the first portion of the solution add dilute NaOH drop-wise until in excess.		
ii)to the second portion of the solution add NH ₃ solution drop-wise until in excess		
iii)to the third portion, of the solution add 2-3 drops of potassium iodide solution.		
c) Wash the residue from (b) with distilled water and add dilute HCl until no further change. Divide the solution into three portions		
i)to the first add dilute NaOH solution drop-wise until in excess		
ii)to the second portion add dilute ammonia solution drop-wise until in excess		
Use the 3 rd portion to carry out a test of your own to confirm the cation present.		

- c) Identify
- the cation in D.....
 - the anion in D.....

Task 7

You are provided with substance Q, which contains two cation and one anion. carry out the following tests on Q and Identify any gas(es) evolved. Record your observations and deductions in the spaces provided.

Test	Observation	Deduction
a) Heat two spatula endful of Q in a dry test tube until there is no further change		
b) Dissolve two spatula end-full of Q in about 8cm ³ of distilled water, divide the resultant solution into five portions		
i)to the first portion of the solution add dilute NaOH drop-wise until in excess.		
ii)to the second portion of the solution add NH ₃ solution drop-wise until in excess		
iii)to the third portion, of the solution add 2-3 drops of potassium hexacyanoferrate(II) solution.		
iv)to the fourth portion add 2-3 drops of lead(II) nitrate solution.		
v)to the fifth portion add dilute nitric acid followed by barium nitrate solution		

- d) Identify
- the cation in Q.....
 - the anion in Q.....

Task 8

You are provided with substance K, which contains two cation and two anions. carry out the following tests on K and to the cations and anions in K. Identify any gas(es) that may be evolved. Record your observations and deductions in the table below.

Test	Observation	Deduction
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a) Heat strongly a spatula endful of K in a dry test tube.		
b) Dissolve two spatula end-full of K in about 5cm ³ of water and to the solution add NaOH drop-wise until in excess, filter keep both residue and filtrate.		
c) to the filtrate add HNO ₃ drop-wise until it is just acidic. Divide into six parts		
i)to the 1 st part add NaOH drop-wise until in excess		
ii)to the 2 nd part add ammonia solution dropwise till in excess.		
iii)to the 3 rd part add solid NH ₄ Cl, followed by 2-3 drops of disodium hydrogen phosphate and excess NH ₃ solution		
iv)to the 4 th part of the acidified filtrate, add 2-3 drops of Pb(NO ₃) ₂ solution		
v)to the first 5 th part of the acidified filtrate, add 2-3 drops of silver nitrate solution.		
vi)to the 6 th part add 2-3 drops of Ba(NO ₃) ₂ solution		
e) Wash residue with water dissolve dilute H ₂ SO ₄ . Divide solution into three parts. i)to 1 st part of the acid solution add NaOH solution drop-wise until in excess.		
ii)to 2 nd part of the acid solution, add NH ₃ solution drop-wise until in excess.		

iii) use to the 3rd portion to confirm one of the cations.

- f) Identify
- the cation in K.....
 - the anion in K.....

Task 9

You are provided with substance Y, which contains two cation and two anion.

You are required to identify the cations and anions in Y

Carry out the following tests on Y and Identify any gas (es) evolved. Record your observations and deductions in the table below.

Test	Observation	Deduction
a) Heat strongly a spatula endful of K in a dry test tube.		
b) shake two spatula end-full of Y with about 3-4cm ³ of water. Filter, keep both filtrate and residue		
c) Divide the filtrate into 4 portions. (i) to the first portion, add NaOH drop-wise till in excess		
ii) to the second portion add NH ₃ drop-wise until in excess.		
iii) to the 3 rd part add 2-3 drops of potassium chromate followed by excess NaOH solution.		
iv) Use 4 th part to carry out own test to confirm anion in the filtrate.		
d) Wash the residue with water. Then add dilute HCl till it dissolves. Divide the resultant solution into 3 parts		

i)to the first part add sodium hydroxide solution drop-wise till in excess		
ii)to the second part, add aqueous ammonia solution dropwise till in excess.		
iii)to 3 rd part add 2-3 drops of potassium chromate followed by excess NaOH solution.		

c) Identify

i) the cation in Y.....

ii) the anion in Y.....

Task 10

You are provided with substance X, which contains two cation and two anions.

You are required to identify the anions and cations in X.

Carry out the following tests on X and Record your observations and deductions in the table below.

Test	Observation	Deduction
a) Heat one spatula endful of X in a dry test tube.		
(b)Put two spatula end-full of X in a test tube. Add about 5cm ³ of water, shake well and filter. keep both residue and filtrate.		
(c)Divide the filtrate into five portions (i)To the first portion of the filtrate add NaOH dropwise until in excess.		
ii)to the second portion of filtrate, add ammonia drop-wise until in excess		
iii)to the third portion of the filtrate, add 2-3 concentrated HNO ₃ , followed by potassium thiocyanate solution.		
iii)to the fourth portion add Lead(II) nitrate solution and warm		

(V) Use the 5 th portion to carry out own test to confirm anion present		
(d) Wash the residue with a little water. Transfer into a test tube and dissolve into dilute hydrochloric acid. Divide the solution into three portions.		
(i) To the first portion of the solution, add dilute sodium hydroxide solution drop-wise until excess.		
(ii) To the 2 nd portion of the solution, add NH_3 solution drop-wise until in excess		
(iii) Use the third portion to carry out a test of your own choice to confirm the cation in the residue.		

(f) Identify :

(i) the cation in X.....

(ii) the anion in X.....

Task 11

You are provided with substance M, which contains one cation and one anion.

Carry out the following tests on M and identify any gas(es) evolved.

Record your observations and deductions in the spaces provided.

Test	Observation	Deduction
a) Heat spatula endful of M strongly in a dry test tube.		
(b) To a spatula end-full of M, add 2-3 drops of conc. Sulphuric acid. heat the mixture.		
(c) Dissolve two spatula endful of M in about 5cm^3 of water and divide the solution into four parts		
(i) To the first part add dilute NaOH drop-wise until in excess		

(ii) To the second part, add dilute ammonia solution drop-wise until in excess.		
(iii) To the fourth part of the solution, add 2-3 drops of lead(II) nitrate solution and warm the mixture.		

(d) Identify:

(i) the cation.....

(ii) the anion in M.....

Task 12

You are provided with substance N, which contains two cation and two anions. Carry out the following tests on N to identify the cations and anions in it. Identify any gas(es) evolved.

Record your observations and deductions in the spaces provided.

Test	Observation	Deduction
a) Heat strongly one spatula endful of N in a dry test tube.		
(b) To one spatula endful of N in a test tube, add 2-3 drops of concentrated sulphuric acid.		
(c) To two spatula endful of N, add about 5 drops of water. Shake vigorously and filter. keep both residue and filtrate.		
(d) Divide the filtrate into six portions. (i) To the first portion of the filtrate, add dilute sodium hydroxide solution drop-wise until in excess		
(ii) To the second portion of the filtrate, add aqueous ammonia solution drop-wise until in excess.		
(iii) To the third portion of filtrate add a spatula endful of solid ammonium chloride followed by 3-4 drops of disodium hydrogen phosphate solution and then ammonia solution drop-wise until in excess.		

(iv) To the fourth portion, add 2-3 drops of potassium chromate(VI) solution followed by 2-3 drops of NaOH solution.		
(v) To the fifth portion of the filtrate, add 2-3 drops of lead(II) nitrate solution followed by dilute nitric acid		
(vi) Use the sixth portion of the filtrate to carry out a test of your own choice to confirm one of the anions in N		
(e) Wash the residue with a little water. Transfer the residue into a test tube and dissolve in dilute nitric acid. Divide the solution into three portions.		
(i) To the first portion of the solution, add dilute sodium hydroxide solution drop-wise until in excess.		
(ii) To the second portion of the solution, add aqueous ammonia solution drop-wise until in excess.		
(iii) Use the third portion of the solution to carry out a test of your own choice to confirm one of the cations in the solution.		

(f) Identify:

(i) the cation in N.....

(ii) the anion in N.....

Task 13

You are provided with substance P, which contains two cation and two anions.

Carry out the following tests on M and identify any gas(es) evolved.

Record your observations and deductions in the spaces provided.

Test	Observation	Deduction
(a) Heat spatula endful of P strongly in a dry test tube.		
(b) To a spatula end-full of P, in at test tube, add about 5cm ³ of water, shake and filter. Keep both		

residue and filtrate. Divide the filtrate into five parts.		
(i)To the first part of the solution, add dilute sodium hydroxide drop-wise until in excess		
(ii)To the second part of the solution, add dilute ammonia solution drop-wise until in excess		
(iii)Use the third part to carry out a test of your own choice to confirm one of the cations in P		
(iv)To the fourth part of the filtrate, add 2-3 drops of lead(II) nitrate solution.		
(v)Use the fifth part of the filtrate to carry out a test of your own to confirm one of the anions in P		
(c)Wash the residue and dissolve the solution into four parts.		
(i)To the first part of the solution, add dilute sodium hydroxide solution drop-wise until in excess		
(ii)To the second part, add dilute ammonia solution drop-wise until in excess.		
(iii)To the third part add 2-3 drops of potassium thiocyanate.		
(iv)Use the fourth part of the solution to carry out a test of your own choice to identify one of the cations in P.		

(d) Identify:

(i) the cations in P.

(ii) the anions in P.

Task 14

You are provided with substance Z, which contains three cation and one anion.

Carry out the following tests on Z to identify the cations and anions in it. Identify any gas(es) evolved.

Record your observations and deductions in the spaces provided.

Test	Observation	Deduction
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a) Heat a spatula endful of Z in a dry test tube until there is no further change.		
(b) Shake two spatula ends-full of Z with about 3cm ³ of water. Add dilute sodium hydroxide solution to the mixture dropwise until in excess. Warm and filter. keep both residue and filtrate		
(c) To the filtrate, add dilute nitric acid drop-wise until the solution is just acidic. Divide the acidic solution into six parts.		
(i) To the first portion of the acidic solution, add dilute NaOH drop-wise until in excess. Warm the mixture.		
(ii) To the second part of the solution, add dilute ammonia solution drop-wise until in excess.		
(iii) To the third part of acidic solution, add 2-3 drops of KI solution		
(iv) To the fourth part of acidic solution, add 2-3 drops of litmus solution followed by NH ₃ solution drop-wise until in excess.		
(v) To the fifth part, add 2-3 drops of lead(II) ethanoate solution.		
(vi) Use the sixth part to carry out a test of your own choice to confirm the anions in Z		

(d)Wash the residue with water and dissolve it in dilute HCl. Divide the solution into three parts.		
(i)To the first part of the acidic solution, add dilute sodium hydroxide solution drop-wise until in excess.		
(ii)To the second part of the acidic solution, add dilute ammonia solution drop-wise until in excess.		
(iii)To the third part of the acidic solution, add 2-3 drops of potassium hexacyanoferrate (II) solution.		

(d) (i) the anion in Z:

(ii) the cations in Z:

Task 15

You are provided with substance Y, which contains two cation and two anions.

Carry out the following tests on Y to identify the cations and anions in it. Identify any gas (es) evolved.

Record your observations and deductions in the spaces provided.

Test	Observation	Deduction
a) Heat one spatula endful of Y in a dry test tube.		
(b)Place two spatula ends-full of Yin a test tube, add 5cm ³ of water. Shake and filter. Keep both residue and filtrate. Divide the filtrate into five portions.		
(i)To the first portion of the filtrate, add dilute sodium hydroxide solution drop-wise until in excess.		

(ii)To the second portion of the filtrate, add dilute ammonia solution.		
(iii)To the third portion of the filtrate, add few drops of potassium hexacyanoferrate (II) solution.		
(iv)To the fourth portion of the filtrate, add 2-3 drops of lead(II) nitrate solution and warm.		
(v)Use the fifth portion of the filtrate to confirm one of the anions in Y.		
(c)Wash the residue with water. Heat a small portion of the residue strongly in a dry test tube.		
(d)Transfer the rest of the residue to a test tube and dissolve it in dilute HCl. Divide the solution into three parts		
(i)To the first part of the solution, add dilute sodium hydroxide solution until in excess		
(ii)To the second part of the resultant solution, add dilute ammonia solution drop-wise until in excess.		
(iii)To the third part of the solution, add 2-3 drops of potassium thiocyanate (II) solution.		

- (e) Identify the ions in substance Y.
- (i) cations
- (ii) anions

ORGANIC QUALITATIVE ANALYSIS

Organic qualitative analysis involves experiments leading to determining the nature of the organic sample. The nature of an organic compounds is described considering the following:

- Whether aliphatic or aromatic.
- Functional group/homologous series (i.e. alcohol, aldehyde, ketone, carboxylic acid, phenol etc.)
- Class, that is 1°, 2° or 3° (where applicable).
- Ratio of C:H i.e. saturation.
- Presence of methyl group attached to functional group carrying carbon (where applicable).

Tests on organic samples

Burning/combustion

OBSERVATION	DEDUCTION
Burns with non-sooty flame	Aliphatic compound
Burns with sooty flame	Aromatic compound

Note: a yellow non sooty flame indicates low C:H ratio (or saturated compound)

Solubility in water

If unknown is miscible with water, test the resultant solution with litmus paper

Observation	Deduction
Miscible with water, resultant solution has no effect on litmus paper	Alcohol, ketone or aldehyde
Miscible with water, resultant solution turns blue litmus paper to red	Carboxylic acid or salt of an amine (also phenol, in case flame was sooty)
Miscible with water, resultant solution turns red litmus paper to blue	Amine or salt of carboxylic acid

Note:

Aromatic carboxylic acids are slightly soluble in cold water but dissolve on heating.

Phenols are slightly soluble giving slightly acidic solutions.

Solubility in NaOH solution

(If compound was immiscible with water)

Observation	Deduction
dissolve in sodium hydroxide solution	Phenol or carboxylic acid

Solubility in HCl(aq)

Observation	Deduction
Soluble in hydrochloric acid solution	Amine

Action of sodium hydrogen carbonate or sodium carbonate (powder or solution)

Observation	Deduction
Effervescence (or bubbles of a colourless gas)	Carboxylic acid
No observable change	Carboxylic acid absent

Alcohol / Ethanoic acid followed by cone. H₂SO₄ and warm

Observation	Deduction
Pleasant fruit smell	Ester formed. Thus, carboxylic acid (or alcohol)
No observable change	Carboxylic acid (or alcohol) absent

Action of acidified potassium dichromate

Observation	Deduction
Dichromate solution turns from orange to green	Primary alcohol, secondary alcohol or aldehyde
No observable change	Primary alcohol, secondary alcohol or aldehyde are absent

Action of sodium metal

Observation	Deduction
Bubbles of a colourless gas, burns with a pop sound	Alcohol

Brady's reagent (2, 4-dinitrophenyl hydrazine)

Observation	Deduction
Yellow precipitate	Ketone or aldehyde
No observable change	Carbonyl compound absent

Sodium hydrogen sulphite solution

Observation	Deduction
White precipitate	Ketone or aldehyde

Lucas reagent (Anhydrous Zinc Chloride and Cone. HCl)

Observation	Deduction
Cloudy solution formed immediately	Tertiary alcohol
Cloudy solution formed after 5 minutes	Secondary alcohol
No observable change	Primary alcohol

Tollen's Reagent (Ammoniacal silver nitrate solution)

Observation	Deduction
Silver mirror deposits	Aldehyde present (if carboxylic acid, then <u>Methanoic acid</u>)
No observable change	Aldehyde absent

Note: Fehling's solution gives brown precipitate with aldehydes

Iodine solution and solution (iodoform)

Observation	Deduction
Yellow precipitate	Methyl group attached to a carbonyl carbon ($\text{CH}_3\text{CO}-$ or CH_3CHO) or $\text{CH}_3\text{C}(\text{OH})\text{H}$

Note: ethanol is the only primary alcohol that gives positive iodoform test.

Iron(II) chloride solution

Observation	Deduction
Violet colouration	Phenol
No observable change	Phenol absent

Bromine water

Observation	Deduction
White precipitate	Phenol (also aromatic amine, in case solution was alkaline)

Hydrochloric acid and sodium nitrite

Observation	Deduction
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Bubbles of colourless gas and a colourless solution	Primary amine
Yellow oily liquid	Secondary amine
No observable change	Tertiary amine (2° and 1° amine absent)

Copper (II) sulphate solution

Observation	Deduction
Deep blue solution	Aliphatic amine
Green precipitate	Phenylamine

Conc. HCl

Observation	Deduction
White precipitate	Aromatic amine

Nitrous acid, 2-Naphthol in sodium hydroxide solution

Observation	Deduction
Red dye	Primary aromatic amine

NOTE:

Salicylic acid is an aromatic organic compound containing both a phenol group and a carboxyl group. It therefore gives a positive phenol test with neutral iron (III) chloride (i.e. violet colouration), and positive test for carboxylic acid (i.e. esterification or effervescence with Sodium carbonate).

Commenting

- Aliphatic (or aromatic)
- Low C:H ratio i.e. saturated (or high C:H ratio i.e. unsaturated)
- Functional group: alcohol, aldehyde, ketone, carboxylic acid or phenol
- Primary or secondary or tertiary (where applicable)
- Methyl carbonyl or CH₃C(OH)H- (where applicable)

Practical Tasks on Qualitative Organic Analysis

Task 1

You are provided with an organic substance **A**. You are required to determine the nature of **A**.

Carry out the following tests on **A** and record your observations and deductions in the table below.

Test	Observations	Deductions
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(a) Burn a small amount of A on a crucible lid or spatula end		
(b) To 1cm ³ of A, add about 1cm ³ of water, shake and test the mixture with litmus		
(c) To 5 drops of A, add 2-3 drops of Brady's reagent.		
(d) To about 2cm of A, add about 1cm ³ of ethanoic acid followed by a few drop of conc. H ₂ SO ₄ . Heat the mixture the pour into a beaker containing water.		
(e) To 5 drops of A, add 2-3 drops of acidified potassium dichromate solution and heat. (keep this solution for next test)		
(f) To the solution formed in (d), add 2-3 drops of Brady's reagent.		
(g) To about 0.5cm ³ of A, add 2-3 drops of Lucas' reagent.		
(h) To about 0.5cm ³ of A, add 2-3 drops of sodium hydroxide solution followed by iodine solution until the solution is pale yellow. Warm the mixture and allow to stand.		

(i) Comment on the nature of **A**.

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Task 2

You are provided with an organic substance **B**. You are required to determine the nature of **B**.

Carry out the following tests on **B** and record your observations and deductions in the table below.

Test	Observations	Deductions
(a) Burn a small amount of B on a crucible lid or spatula end		
(b) To 1cm ³ of B, add about 2cm ³ of water, shake and test the mixture with litmus. Divide the mixture into two parts		
(c) To the first part, add 2-3 drops of Brady's reagent		
(d) To the second part, add 2-3 drops of acidified potassium dichromate solution. Heat the mixture and divide the mixture into two parts.		
(e) To the first part of the solution, add 2-3 drops of Brady's reagent.		
(f) To the second part, of the solution, add 2-3 drops of Tollen's reagent and warm.		
(g) To about 0.5cm ³ of B, add 2-3 drops of Lucas' reagent.		
(f) To about 0.5cm ³ of B, add 2-3 drops of sodium hydroxide solution followed by iodine solution until the solution is pale yellow. Warm the mixture and allow to stand.		

(h) Comment on the nature of B.

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Task 3

You are provided with an organic substance **C**. You are required to determine the nature of **C**.

Carry out the following tests on **C** and record your observations and deductions in the table below.

Test	Observations	Deductions
(a) Burn a small amount of C on a crucible lid or spatula end.		
(b) To 1cm ³ of C, add about 1cm ³ of water, shake and test the mixture with litmus		
(c) To 5 drops of C, add 2-3 drops of Brady's reagent.		
(d) To about 0.5cm ³ of C, add about 1cm ³ of Tollen's reagent, warm and allow the mixture to stand.		
(e) To about 0.5cm ³ of C, add 2-3 drops of sodium hydroxide solution followed by iodine solution until the solution is pale yellow. Warm the mixture and allow to stand.		

(h) Comment on the nature of C.

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Task 4

You are provided with an organic substance **D**. You are required to determine the nature of **D**.

Carry out the following tests on **D** and record your observations and deductions in the table below.

Test	Observations	Deductions
(a) Burn a small amount of D on a crucible lid or spatula end		
(b) To 1cm ³ of D, add about 1cm ³ of water,		

shake and test the mixture with litmus		
(c) To 5 drops of D, add 2-3 drops of Brady's reagent.		
(d) To about 0.5cm ³ of D, add about 1cm ³ of Tollen's reagent, warm and allow the mixture to stand.		
(e) To about 0.5cm ³ of D, add 2-3 drops of sodium hydroxide solution followed by iodine solution until the solution is pale yellow. Warm the mixture and allow to stand.		

(f) Comment on the nature of **D**.

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Task 5

You are provided with an organic substance **E**. You are required to determine the nature of **E**.

Carry out the following tests on **E** and record your observations and deductions in the table below.

Test	Observations	Deductions
(a) Burn a small amount of E on a crucible lid or spatula end		
(b) To 1cm ³ of E, add about 1cm ³ of water, shake and test the mixture with litmus. Divide the resultant solution into two portions.		
(c) To the first portion of the solution, add 1cm ³ of sodium hydrogen carbonate solution.		
(d) To second portion of the solution, add 2-3 drops		

of 2, 4-dinitrophenyl hydrazine.		
(e) To about 0.5cm ³ of E, add about 1cm ³ of Tollen's reagent, warm and allow the mixture to stand		

(f) From your results above, deduce the nature of compound **E**.

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Task 6

You are provided with an organic substance **F**. You are required to determine the nature of **F**.

Carry out the following tests on **F** and record your observations and deductions in the table below.

Test	Observations	Deductions
(a) Burn a small amount of F on a crucible lid or spatula end		
(b) Transfer one spatula endful of F to a test tube containing 3 cm ³ of water, warm the mixture and test with litmus. Divide the warm solution into three parts.		
(i) To the first part of the warm solution, add about 1cm ³ of sodium hydroxide solution.		
(ii) To the second part of the warm solution, add 2-3 drops of sodium carbonate solution.		
(iii) To the third part, add 2-3 drops of Iron (III) chloride solution.		

(c) Comment on the nature of **F**.

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Task 7

You are provided with an organic substance **G**. You are required to determine the nature of **G**.

Carry out the following tests on **G** and record your observations and deductions in the table below.

Test	Observations	Deductions
(a) Burn a small amount of G on a crucible lid or spatula end		
(b) Transfer one spatula endful of G to a test tube containing 3cm ³ of water, warm the mixture and test with litmus. Divide the warm solution into three parts. (i) To the first part of the warm solution, add 2-3 drops of Brady's reagent.		
(ii) To the second part of the warm solution, add 2-3 Tollen's reagent and allow to cool.		
(iii) To about 0.5cm ³ of D , add 2-3 drops of sodium hydroxide solution followed by iodine solution until the solution is pale yellow. Warm the mixture and allow to stand.		

(c) Comment on the nature of **G**.

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Task 8

You are provided with an organic substance **H**. You are required to determine the nature of **H**.

Carry out the following tests on **H** and record your observations and deductions in the table below.

Test	Observations	Deductions
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(a) Burn a small amount of H on a crucible lid of spatula end.		
(b) To 5 drops of H, add about 1cm ³ of water and shake. Test the mixture with litmus.		
(c) To 5 drops of H, add 2-3 drops of acidified potassium manganate(VII) solution and warm.		
(d) To 5 drops of H, add 1cm ³ of iodine solution, followed by dilute sodium hydroxide drop-wise until the mixture is pale yellow. Warm, then cool under a tap.		
(e) To 5 drops of H, add 2-3 drops of Brady's reagent.		
(f) Carry out a test of your own choice to confirm the functional group in H.		

(g) Comment on the nature of H.

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Task 9

You are provided with an organic substance I. You are required to determine the nature of I.

Carry out the following tests on I and record your observations and deductions in the table below.

Test	Observations	Deductions
(a) Burn a small amount of I on a crucible lid of spatula end.		
(b) To 5 drops of I, add about 1cm ³ of water and warm. Test the mixture with litmus.		

(c) To 2cm ³ of I, add about 2cm ³ of sodium hydroxide solution.		
(d) To 2cm ³ of I, add 3 drops of iron (III) chloride solution.		
(e) To 2cm ³ of I, add half a spatula of sodium hydrogen carbonate.		
(f) To-about 2cm ³ of I, add 2cm ³ of ethanol followed by 3 drops concentrated sulphuric acid and boil.		

(g) Comment on the nature of I.

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