ADVANCED CRYSTAL CHEMISTRY (By: Kenneth Opio Ojok)

DRY – QUANTITATIVE ANALYSIS (FOR S.6 Candidates)

a) COMPLEX MIXTURES (Double Indicator Titrations)

Experiment 1

You are provided with the following:

FA1 which is a solution of **0.3M** hydrochloric acid.

FA2 which is a mixture of potassium hydroxide and potassium carbonate solution.

Aim:

To determine the:

(i) Concentrations of potassium carbonate in gdm^{-3}.
(ii) Percentage of potassium hydroxide in the FA2 mixture.
(K =39, C = 12, O = 16, H =1)

Procedure:

Pipette $25cm^3$ or $20cm^3$ of FA2 into a clean conical flask. Add 2 to 3 drops of phenolphthalein indicator and titrate with FA1 from the burette until the end point is reached. Record your results in **table 1** below. Then continue with the titration by adding 2 to 3 drops of methyl orange indicator to the resultant solution and continue with FA1 from the burette until the end point is reached. Record your results in **table 2** below. Repeat the titration until you obtain consistent results.

Volume of pipette used
……cm^3

Table 1 range:	**Table 2 range**:
$20.00cm^3$ to $20.30cm^3$ if $25cm^3$ is used	**$10.00cm^3$ to $10.30cm^3$** if $25cm^3$ is used
$15.00cm^3$ to $15.30cm^3$ if $20cm^3$ is used	**$9.00cm^3$ to $9.30cm^3$** if $20cm^3$ is used

Burette readings	**Table 1** With phenolphthalein indicator			**Table 2** With methyl orange indicator		
Final burette reading / cm^3						
Initial burette reading/ cm^3						
Volume of FA1 used / cm^3						

Average Titre volume of FA1 used for table 1

……cm^3

Average Titre volume of FA1 used for table 2

……cm^3

Theory: (1) With phenolphthalein indicator:

$KOH_{(aq)} + HCl_{(aq)} \longrightarrow KCl_{(aq)} + H_2O_{(l)}$

$K_2CO_{3\,(aq)} + HCl_{(aq)} \longrightarrow \mathbf{KHCO_{3(aq)}} + KCl_{(aq)}$

(2) With Methyl orange indicator:

$\mathbf{KHCO_{3\,(aq)}} + HCl_{(aq)} \longrightarrow KCl_{(aq)} + CO_{2(g)} + H_2O_{(l)}$

Questions:

a) Determine the volume of hydrochloric acid in FA1 required for complete neutralization of:
   (i) Potassium carbonate:

   ……...cm^3

   (ii) Potassium hydroxide:

   …...……...cm^3

b) Calculate the concentration of:

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(i) Potassium carbonate in FA2 in grams per litre.

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(ii) Potassium hydroxide in FA2 in gdm^{-3}

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c) Determine the percentage of potassium hydroxide in FA2 mixture.

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Experiment 2

You are provided with the following:

FA1 which is a solution of **0.1M** Sulphuric acid.

FA2 which is a mixture of sodium hydroxide and sodium carbonate solution.

Aim:

To determine the:

(i) Concentrations of sodium carbonate in gdm^{-3}.
(ii) Percentage of sodium hydroxide in the FA2 mixture.
(**Na =23, C = 12, O = 16, H =1**)

Procedure 1:

Pipette $25cm^3$ or $20cm^3$ of FA2 into a clean conical flask. Add 2 to 3 drops of phenolphthalein indicator and titrate with FA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in **table 1** below.

Table 1 range: $17.00cm^3$ to $17.20cm^3$ if $25cm^3$ is used OR $14.00cm^3$ to $14.20cm^3$ if $20cm^3$ is used

Volume of pipette used:
……cm^3

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Table with phenolphthalein indicator

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

Average Titre volume of FA1 used

……cm^3

Procedure II

Pipette $25cm^3$ or $20cm^3$ of FA2 into a clean conical flask. Add 2 to 3 drops of methyl orange indicator and titrate with FA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in **table 2** below.

Table 2 range: 25.00cm^3 to 25.20cm^3 if 25cm^3 is used OR 20.00cm^3 to 20.20cm^3 if 20cm^3 is used

Volume of pipette used:

……cm^3

Table with methyl orange indicator

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

Average Titre volume of FA1 used

……cm^3

Theory: Table 1With phenolphthalein indicator $2NaOH_{(aq)} + H_2SO_{4\,(aq)} \longrightarrow Na_2SO_{4\,(aq)} + 2H_2O_{(l)}$	**Theory**: Table 2 With methyl orange indicator $2NaOH_{(aq)} + H_2SO_{4\,(aq)} \longrightarrow Na_2SO_{4\,(aq)} + 2H_2O_{(l)}$
$2Na_2CO_{3\,(aq)} + H_2SO_{4\,(aq)} \longrightarrow 2NaHCO_{3(aq)} + Na_2SO_{4(aq)}$	$2Na_2CO_{3\,(aq)} + H_2SO_{4\,(aq)} \longrightarrow Na_2SO_{4(aq)} + CO_{2(g)} + 2H_2O_{(l)}$

Questions:

a) Determine the volume of hydrochloric acid in FA1 required for complete neutralization of:

(i) Sodium carbonate:

……cm^3

(ii) Sodium hydroxide:

……cm^3

b) Calculate the concentration of:

(i) Sodium carbonate in FA2 in grams per litre.

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(ii) Sodium hydroxide in FA2 in gdm^{-3}

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c) Determine the percentage of potassium hydroxide in FA2 mixture.

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b) REDOX TITRATION

(i) **That which involves acidified potassium manganate (VII) solution**.

Experiment 3:

You are provided with the following:

FA1 which is a solution of **potassium manganate (VII), $KMnO_4$**

FA2 which is a solution of 2**M** Sulphuric acid.

Solid H which are crystals of sodium oxalate, $Na_2C_2O_4$

Aim:

To determine the Concentrations of:

(i) sodium oxalate in FA3 in moles per litre.

(ii) Potassium manganate (VII) in FA1 in moles per litre.

Theory:

Manganate (VII) ions are reduced to manganese (II) ions in acidic medium according to the equation below.

$$MnO_4^-{}_{(aq)} + 8H^+{}_{(aq)} + 5e^- \longrightarrow Mn^{2+}{}_{(aq)} + 4H_2O_{(l)}$$

Oxalate ions are oxidized to carbon dioxide gas according to the equation below.

$$C_2O_4^{2-}{}_{(aq)} \longrightarrow CO_{2(g)} + 2e^-$$

Procedure I:

1. Weigh accurately 1.6g of H into a clean Beaker. Add about 100cm^3 of distilled water and stir well to dissolve. Transfer the solution into a 250cm^3 volumetric flask and make it up to the mark with distilled water. label this solution FA3.
2. Pipette 20 or 25cm^3 of FA3 into a clean conical flask. Using a measuring cylinder transfer an equal volume of FA2 into a conical flask containing FA3. Heat the solution mixture to about 60^{o}C and immediately titrate the hot solution mixture with FA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below:

Mass of beaker + H ……..g
Mass of beaker a lone……..g
Mass of H ……..g
Volume of pipette used ……cm^3

Table I range:

22.00cm^3 to 22.40cm^3 if 25cm^3 is used
17.50cm^3 to 17.90cm^3 if 20cm^3 is used.

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

Values used to calculate average volume =
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Average volume of FA1 used =
……cm^3

Questions:

a) Write the overall redox equation between acidified manganate (VII) ions and oxalate ions.

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b) Calculate the concentration of:

(i) Sodium oxalate in FA3 in mole per litre.

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(ii) Potassium manganate (VII) in FA1 in moles per litre.

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Experiment 4:

You are provided with the following:

FA1 which is a solution of **potassium manganate (VII), $KMnO_4$**

FA2 which contains 11.5g of **$FeSO_4.xH_2O$** in $500cm^3$ solution.

FA3 which is a solution of 2**M** Sulphuric acid.

Solid **W** which are crystals of sodium oxalate, $Na_2C_2O_4$

Aim:

To determine the Concentrations of:

(i) Potassium manganate (VII) in FA1 in moles per litre.

(ii) Value of x in **$FeSO_4.xH_2O$**

(Na =23, C =12, H =1, Fe =56, S =32)

Theory:

Manganate (VII) ions react with Fe^{2+} and $C_2O_4^{2-}$ according to the following equations below:

$MnO_4^-{}_{(aq)} + 8H^+{}_{(aq)} + 5Fe^{2+}{}_{(aq)} \longrightarrow Mn^{2+}{}_{(aq)} + 5Fe^{3+}{}_{(aq)} + 4H_2O_{(l)}$

$MnO_4^-{}_{(aq)} + 16H^+{}_{(aq)} + 5C_2O_4^{2-}{}_{(aq)} \longrightarrow Mn^{2+}{}_{(aq)} + 8H_2O_{(l)} + 10CO_{2\,(g)}$

Procedure I:

Weigh accurately 1.4g of W into a clean Beaker. Add about $100cm^3$ of distilled water and stir well to dissolve. Transfer the solution into a $250cm^3$ volumetric flask and make it up to the mark with distilled water. label this solution FA4.

Pipette 20 or $25cm^3$ of FA4 into a clean conical flask. Using a measuring cylinder transfer an equal volume of FA3 into a conical flask containing FA4. Heat the solution mixture to about 60^oC and immediately titrate the hot solution mixture with FA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below:

Mass of beaker + W ……...g

Mass of beaker a lone……...…...g

Mass of W ……...…...g

Volume of pipette used ……...cm^3

Table I range:

$23.00cm^3$ to $23.30cm^3$ if $25cm^3$ is used.

$18.00cm^3$ to $18.30cm^3$ if $20cm^3$ is used.

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

Value used to calculate average =

……cm^3

Average volume of FA1 used =

……...cm^3

Procedure II:

Pipette 20 or 25cm^3 of FA2 into a clean conical flask. Using a measuring cylinder transfer an equal volume of FA3 into a conical flask containing FA2 and titrate the solution mixture with FA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below:

Volume of pipette used ……...cm^3

Table II range:

22.80cm^3 to 23.00cm^3 if 25cm^3 is used.

18.40cm^3 to 18.60cm^3 if 20cm^3 is used.

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

Value used to calculate average =

……cm^3

Average volume of FA1 used =

……..……cm^3

Questions:

a) Calculate the concentration of:

(i) Sodium oxalate in FA4 in moles per litre.

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(ii) Potassium manganate (VII) in FA1 in moles per litre.

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(iii) $FeSO_4.xH_2O$ in FA2 in moles per litre.

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b) Determine the value of x in $FeSO_4.xH_2O$.

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Experiment 5:

You are provided with the following:

FA1 which contains 0.675g of **potassium manganate (VII), $KMnO_4$** in 250cm^3 of solution.

FA2 which a solution containing a mixture of iron (II) sulphate heptahydrate, **$FeSO_4.7H_2O$** and diammonium iron (III) sulphate – 12 – water, $(NH_4)_2SO_4Fe_2(SO_4)_3.\ 12H_2O$.

FA3 which is a solution of 2**M** Sulphuric acid.

Solid **Q** which is magnesium metal powder.

Aim:

To determine the percentage of iron (II) sulphate in FA2 mixture.

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

Theory:

Manganate (VII) ions react with Fe^{2+} according to the following equations below:

$$MnO_4^-{}_{(aq)} + 8H^+_{(aq)} + 5Fe^{2+}_{(aq)} \longrightarrow Mn^{2+}_{(aq)} + 5Fe^{3+}_{(aq)} + 4H_2O_{(l)}$$

Magnesium metal powder reacts with iron (III) ions in FA2 mixture in acidic medium according to the equation below. $2Fe^{3+}_{(aq)} + Mg_{(s)} \longrightarrow 2Fe^{2+}_{(aq)} + Mg^{2+}_{(aq)}$

Procedure I

Pipette 20 or 25cm^3 of FA2 into a clean conical flask. Using a measuring cylinder transfer an equal volume of FA3 into a conical flask containing FA2 and titrate the solution mixture with FA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below:

Volume of pipette used ……...cm^3

Table 1 range:

23.50cm^3 to 23.80cm^3 if 25cm^3 is used

18.30cm^3 to 18.50cm^3 if 20cm^3 is used.

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

Value used to calculate average =

……cm^3

Average volume of FA1 used =

……...cm^3

Questions:

a) Determine the concentration of:

(i) potassium manganate (VII) in FA1 $moldm^{-3}$

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(ii) Fe^{2+} in FA2 in $moldm^{-3}$

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Procedure II

Transfer 120cm^3 of FA2 using a measuring cylinder into a clean conical flask. Add 2g of magnesium metal powder followed by 40cm^3 of FA3. Warm the mixture until the solution mixture obtained is almost colourless. Allow the solution mixture to stand and cool. Label this solution FA4.

Pipette 20 or 25cm^3 of FA4 into a clean conical flask. Using a measuring cylinder transfer an equal volume of FA3 into a conical flask containing FA2 and titrate the solution mixture with FA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below:

Volume of pipette used ……...cm^3

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

Value used to calculate average =

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Average volume of FA1 used =

……...cm^3

Questions:

b) Calculate the:

(i) Total number of moles of Fe^{2+} in 160cm^3 of FA4.

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(ii) Total concentration of Fe^{2+} in the FA2 solution used in procedure II in moldm^{-3}

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(iii) Concentration of Fe^{3+} in FA2 solution in moldm^{-3}

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c) Determine the:

(i) Total mass of the iron (II) and iron (III) salt in 1dm^3 of FA2.

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(ii) Percentage by mass of iron (II) salt in FA2 solution

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Experiment 6:

You are provided with the following:

FA1 which contains 3.2g of **potassium manganate (VII), $KMnO_4$** per litre.

FA2 which contains 4.48g of potassium hydroxide per litre

FA4 which is a solution of 2**M** Sulphuric acid.

Solid **E** which is a dibasic compound of the formula **$H_w(C_2O_4)_x.yH_2O$.**

Aim:

To determine the Value of **w, x** and **y** in **$H_w(C_2O_4)_x.yH_2O$.**

Theory:

Solid E readily dissolves in water. In aqueous state, the acidic compound ionizes according to the equation below:

$H_w(C_2O_4)_x.yH_2O_{(aq)} \longrightarrow wH^+_{(aq)} + xC_2O_4^{2-}{}_{(aq)} + yH_2O_{(l)}$

The H^+ ions produced from the acid react with hydroxyl ions from potassium hydroxide according to the equation below:

$H^+_{(aq)} + OH^-_{(aq)} \longrightarrow H_2O_{(l)}$

Acidified manganate (VII) ions from potassium manganate (VII) react with oxalate ions (ethandioate ions) according to the equation below

$2MnO_4^-{}_{(aq)} + 5C_2O_4^{2-}{}_{(aq)} + 16H^+_{(aq)} \longrightarrow 2Mn^{2+}_{(aq)} + 10CO_{2(g)} + 8H_2O_{(l)}$

Procedure I:

Weigh accurately 1.2g of E into a clean Beaker. Add about 100cm^3 of distilled water and stir well to dissolve. Transfer the solution into a 250cm^3 volumetric flask and make it up to the mark with distilled water. label this solution FA3. Pipette 20 or 25cm^3 of FA3 into a clean conical flask. Using a measuring cylinder transfer an equal volume of FA4 into a conical flask containing FA3 and titrate the solution mixture with FA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below:

Mass of beaker + E ……...g

Mass of beaker a lone……...…...g

Mass of E ……...…...g

Volume of pipette used ……...cm^3

Table I range:

23.50cm^3 to 23.80cm^3 if 25cm^3 is used.

18.80cm^3 to 19.00cm^3 if 20cm^3 is used.

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

Value used to calculate average =

……cm^3

Average volume of FA1 used =

……...cm^3

Procedure II

Pipette 20 or 25cm^3 of FA3 into a clean conical flask. Using a measuring cylinder transfer an equal volume of FA4 into a conical flask containing FA3 and heat the solution mixture up to 70^{o}C. Titrate the solution mixture with FA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below:

Volume of pipette used ……...cm^3

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Table II range:
21.00cm³ to 21.20cm³ if 25cm³ is used.
16.80cm³ to 17.00cm³ if 20cm³ is used.

Final burette reading/cm³			
Initial burette reading/cm³			
Volume of FA1 use//cm³			

Value used to calculate average =
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Average volume of FA1 used =
……..cm³

Questions:

a) Calculate the concentration of:

(i) Hydrogen ions, H^+ in FA3 in moles per litre.

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(ii) Oxalate ions, $C_2O_4^{2-}$ in FA3 in moles per litre.

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b) Determine the

(i) mole ratios of **w** to **x.**

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(ii) Value of **y** in $H_w(C_2O_4)_{x.}{}_yH_2O$.

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Experiment 7:

You are provided with the following:

FA1 which is a solution that contains 1.26g of anhydrous sodium sulphite, Na_2SO_3 in 200cm³ of solution.

FA2 which is a solution of **potassium manganate (VII), $KMnO_4$**

2**M** Sulphuric acid solution

Solid F which is an impure ferrous ethanedioate, **FeC_2O_4**

Aim:

To determine the:

(i) Molar concentration of potassium manganate (VII) in FA2.

(ii) Percentage impurity in Ferrous ethanedioate sample.

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Theory:

Manganate (VII) ions react with Fe^{2+}, $C_2O_4^{2-}$ and SO_3^{2-} according to the following equations below:

$MnO_4^-{}_{(aq)} + 8H^+{}_{(aq)} + 5Fe^{2+}{}_{(aq)} \longrightarrow Mn^{2+}{}_{(aq)} + 5Fe^{3+}{}_{(aq)} + 4H_2O_{(l)}$

$MnO_4^-{}_{(aq)} + 16H^+{}_{(aq)} + 5C_2O_4^{2-}{}_{(aq)} \longrightarrow Mn^{2+}{}_{(aq)} + 8H_2O_{(l)} + 10CO_{2\,(g)}$

$2MnO_4^-{}_{(aq)} + 6H^+{}_{(aq)} + 5SO_3^{2-}{}_{(aq)} \longrightarrow 2Mn^{2+}{}_{(aq)} + 5SO_4^{2-}{}_{(aq)} + 3H_2O_{(l)}$

Using a measuring cylinder transfer 20cm^3 of FA1 into a conical flask. Add 10cm^3 of 2M sulphuric acid and titrate the solution mixture with FA2 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below:

Volume of pipette used ……...cm^3

Table I range:

21.20cm^3 to 21.50cm^3 if 25cm^3 is used.

16.20cm^3 to 16.50cm^3 if 20cm^3 is used.

Final burette reading/cm^3			
Initial burette reading/cm^3			
Volume of FA2 use//cm^3			

Value used to calculate average =

……cm^3

Average volume of FA1 used =

……...cm^3

Procedure II

Weigh accurately 1.5g of F into a clean Beaker. Using a measuring cylinder transfer 100cm^3 of 2M sulphuric acid into the beaker containing the solid and stir well to dissolve. Transfer the solution into a 250cm^3 volumetric flask and make it up to the mark with distilled water. label this solution FA3.

Pipette 20 or 25cm^3 of FA3 into a clean conical flask and heat the solution to 70°C and titrate the solution mixture with FA2 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below:

Mass of beaker + F ……...g

Mass of beaker a lone……...…...g

Mass of F ……...…...g

Volume of pipette used ……...cm^3

Table II range:

26.00cm^3 to 26.50cm^3 if 25cm^3 is used.

21.00cm^3 to 21.30cm^3 if 20cm^3 is used.

Final burette reading/cm^3			
Initial burette reading/cm^3			
Volume of FA2 use//cm^3			

Value used to calculate average =

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Average volume of FA2 used =

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Questions:

a) Calculate the:

(i) molarity of potassium manganate (VII) in FA2 (Na =23, S =32, O =16)

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(ii) number of moles of manganate (VII) ions that reacted with sulphite ions in 20 or (25) cm^3 of FA3.

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(iii) number of moles of iron (II) ions in 25 (20) cm^3 of FA3

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b) Determine the:

(i) mass of Ferrous ethanedioate, **FeC_2O_4** in 250cm^3 of FA3. (Fe =56, C =12, O =16)

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(ii) percentage impurity in the ferrous ethanedioate sample.

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(ii) REDOX TITRATION THAT INVOLVE IODOMETRY

Experiment 8:

You are provided with the following:

FA1 which is a solution containing **1.12g** of potassium chromate (VI) in 200cm^3 of solution.

FA2 which is a solution containing 25.0g of the hydrated metal thiosulphate, **$XS_2O_3.nH_2O$** in one litre of solution

FA3 is 10% of potassium iodide solution.

FA4 is 2M sulphuric acid.

Solid T which is an impure potassium iodate, **KIO_3**

Aim:

To determine the:

(i) molarity of the metal thiosulphate in **FA2**

(ii) value of **n** in **$XS_2O_3.nH_2O$**

(iii) percentage purity of potassium iodate in FA5

Theory:

In acidic medium, **CrO_4^{2-}** ions are readily converted into dichromate ions, $Cr_2O_7^{2-}$ according to the equation below:

$2CrO_4^{2-}{}_{(aq)} + 2H^+{}_{(aq)} \longrightarrow Cr_2O_7^{2-}{}_{(aq)} + H_2O_{(l)}$

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In acidic medium, IO_3^- ions and $Cr_2O_7^{2-}$ ions are converted to iodine and chromium (III) ions respectively according to the equations below:

$$IO_3^-{}_{(aq)} + 6H^+{}_{(aq)} + 5I^-{}_{(aq)} \longrightarrow 3I_{2(aq)} + 3H_2O_{(l)}$$

$$Cr_2O_7^{2-} + 14H^+{}_{(aq)} + 6I^-{}_{(aq)} \longrightarrow 2Cr^{3+}{}_{(aq)} + 7H_2O_{(l)} + 3I_{2(aq)}$$

The liberated Iodine is then titrated with thiosulphate ions from the burette, they react according to the equation below

$$2S_2O_3^{2-}{}_{(aq)} + I_{2\,(aq)} \longrightarrow S_4O_6^{2-}{}_{(aq)} + 2I^-{}_{(aq)}$$

Procedure I

Pipette 20 or 25cm^3 of **FA1** into a clean conical flask. Add 30cm^3 of **2M** sulphuric acid followed by 10cm^3 of potassium iodide solution. Titrate the mixture with **FA2** from the burette until the solution just becomes pale yellow, then add 1cm^3 of starch indicator and continue with the titration until the dark – blue solution just turns to a pale (light) blue solution. Repeat the titration until you obtain consistent results. Record your findings in the table 1 below.

Volume of pipette used ……..cm^3

Table I range:

21.30cm^3 to 21.60cm^3 if 25cm^3 is used.

17.30cm^3 to 17.60cm^3 if 20cm^3 is used.

Final burette reading/cm^3			
Initial burette reading/cm^3			
Volume of FA2 use//cm^3			

Value used to calculate average =

……cm^3

Average volume of FA2 used =

……...cm^3

Questions:

a) Calculate the:

(i) number of CrO_4^{2-} ions in FA1 in 20 or 25cm^3 .

(K=39, Cr =24, O =16)

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(ii) molar concentration of thiosulphate ions $S_2O_3^{2-}$ in FA2

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(iii) value of x in **$XS_2O_3.nH_2O$ (M =46, S =32, O =16, H =1)**

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Procedure II

Weigh accurately 1.0g of solid T into a clean conical flask. Add about 150cm^3 of distilled water and stir well to dissolve. Transfer the solution to a 250cm^3 volumetric flask and make up to the mark with distilled water. label this solution FA5.

Transfer 25cm^3 of FA5 into a clean conical flask using a measuring cylinder and add 10cm^3 of 10% potassium iodide solution followed by 10cm^3 of 2M sulphuric acid. Titrate the liberated iodine with FA2 from the burette until the solution just turns to pale yellow. Add 1cm^3 of starch indicator and continue with the titration until the dark – blue solution just turns colourless. Repeat the titration until you obtain consistent results. Record your findings in the table 2 below:

Mass of beaker + T ……...g
Mass of beaker a lone……...…...g
Mass of T ……...…...g
Volume of pipette used ……...cm^3

Table I range:

21.30cm^3 to 21.60cm^3 if 25cm^3 is used.
17.30cm^3 to 17.60cm^3 if 20cm^3 is used.

Final burette reading/cm^3			
Initial burette reading/cm^3			
Volume of FA2 use//cm^3			

Value used to calculate average =
……cm^3

Average volume of FA2 used =
……...cm^3

Questions:

b) Determine the:

(i) number of moles of IO_3 ions in 250cm^3 of FA5.

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(ii) mass of potassium Iodate in 250cm^3 of FA5.

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(iii) percentage purity of potassium iodate in the sample used in FA5 (K =39, I =127, O =16)

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Experiment 9:

You are provided with the following:

FA1 which is a solution containing **1.8g** of potassium dichromate (VI) in 500cm^3 of solution.

FA2 hydrogen peroxide solution

FA3 which is a solution of sodium thiosulphate – 5 – water in one litre of solution

FA4is 10% of potassium iodide solution.

2M sulphuric acid solution.

Aim:

To determine the:

(i) concentration of sodium thiosulphate in FA3 in gdm^{-3}

(ii) volume strength of hydrogen peroxide in FA2.

Theory:

In acidic medium, dichromate (VI) ions and hydrogen peroxide react with iodide ions according to the equations below:

$Cr_2O_7^{2-} + 14H^+_{(aq)} + 6I^-_{(aq)} \longrightarrow 2Cr^{3+}_{(aq)} + 7H_2O_{(l)} + 3I_{2(aq)}$

$H_2O_{2\,(aq)} + 2H^+_{(aq)} + 2I^-_{(aq)} \longrightarrow I_{2\,(aq)} + 2H_2O_{\,(l)}$

Procedure I

Pipette 20 or 25cm^3 of **FA1** into a clean conical flask. Add an equal volume of **2M** sulphuric acid. Titrate the mixture with **FA3** from the burette until the solution just becomes pale yellow, then add 1cm^3 of starch indicator and continue with the titration until the dark – blue solution just turns to a pale (light) blue solution. Repeat the titration until you obtain consistent results. Record your findings in the table 1 below.

Volume of pipette used ……...cm^3

Table I range:

21.30cm^3 to 21.60cm^3 if 25cm^3 is used.

17.30cm^3 to 17.60cm^3 if 20cm^3 is used.

Final burette reading/cm^3			
Initial burette reading/cm^3			
Volume of FA3 use//cm^3			

Value used to calculate average =

……cm^3

Average volume of FA3 used =

……...cm^3

Questions:

a) Determine the molar concentration of:

(i) Potassium dichromium (VI) in FA1.

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(ii) Sodium thiosulphate in FA3.

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PART II Procedure:

Transfer 100cm^3 of FA3 into a clean conical flask. Add 100cm^3 of distilled water, shake well to mix. Label this solution FA5.

Pipette 20 or 25cm^3 of **FA2** into a clean conical flask. Add an equal volume of **FA4** followed by 30cm^3 of 2M sulphuric acid using a measuring cylinder. Leave the mixture to settle for 12 minutes and the titrate the mixture with **FA5** from the burette until the solution just becomes pale yellow, then add 1cm^3 of starch indicator and continue with the titration until the dark – blue solution just turns to a pale (light) blue solution. Repeat the titration until you obtain consistent results. Record your findings in the table 1 below.

Volume of pipette used ……...cm^3

Table II range:

16.60cm^3 to 16.80cm^3 if 25cm^3 is used.

13.30cm^3 to 13.50cm^3 if 20cm^3 is used.

Final burette reading/cm^3			
Initial burette reading/cm^3			
Volume of FA3 use//cm^3			

Value used to calculate average =

……cm^3

Average volume of FA3 used =

……...cm^3

Questions:

b) Calculate the:

c) Determine the molar concentration of:

(i) Sodium thiosulphate in FA5

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(ii) Hydrogen peroxide in FA2.

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(iii) Volume strength of hydrogen peroxide in the FA2 solution. (**NB**: *Volume strength is the volume of oxygen gas liberated by 1cm^3 of hydrogen peroxide solution; 1 mole of a gas occupies 24dm^3 at room temperature*)

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BACK TITRATION

Experiment 10:

You are provided with the following

FA1 which is 0.1M potassium carbonate solution

FA2 which is 0.1M potassium hydroxide solution.

FA3 which approximately 1M sulphuric acid

Solid M which is a metal oxide, XO

Aim:

To determine the:

(i) molar concentration of sulphuric acid in FA3 in moles per litre.

(ii) value of X in the metal oxide, XO.

Theory:

The metal oxide, XO reacts with sulphuric acid which is in excess according to the equation.

$$XO_{(s)} + H_2SO_{4(aq)} \longrightarrow XSO_{4(aq)} + H_2O_{(l)}$$

The excess, unreacted sulphuric acid is then reacted with potassium hydroxide solution according to the equation below:

$$2KOH_{(aq)} + H_2SO_{4(aq)} \longrightarrow K_2SO_{4(aq)} + H_2O_{(l)}$$

PART I Procedure

Transfer 20cm^3 of **FA3** into a clean conical flask. Add 100cm^3 of distilled water, shake well to mix. Label this solution **FA4**.

Pipette 20 or 25cm^3 of **FA1** into a clean conical flask. Add 2 or 3 drops of methyl orange indicator and titrate the mixture with **FA4** from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your findings in the table 1 below.

Volume of pipette used ……...cm^3

Table I range:

14.80cm^3 to 15.00cm^3 if 25cm^3 is used.

11.80cm^3 to 12.00cm^3 if 20cm^3 is used.

Final burette reading/cm^3			
Initial burette reading/cm^3			
Volume of FA3 use//cm^3			

Value used to calculate average =

……cm^3

Average volume of FA3 used =

……...cm^3

Questions:

a) Calculate the:

(i) number of moles of sulphuric acid in the 120cm^3 of FA4

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(ii) molar concentration of sulphuric acid in FA3 in moles per litre.

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PART II Procedure

Weigh accurately 3.0g of solid M into a clean conical flask. Add 30cm^3 of FA3 and stir well to dissolve. (You may warm gently as you stir to dissolve if it is necessary). Transfer the solution to a 250cm^3 volumetric flask and make up to the mark with distilled water. label this solution FA5.

Mass of beaker + M ……...g
Mass of beaker a lone……...…...g
Mass of M ……...…...g

Pipette 20 or 25cm^3 of FA2 into a clean conical flask. Add 2-3 drops of methyl orange indicator and titrate with FA5 from burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your findings in the table 1 below.

Volume of pipette used ……...cm^3

Table II range:

20.50cm^3 to 20.80cm^3 if 25cm^3 is used.
16.50cm^3 to 16.80cm^3 if 20cm^3 is used.

Final burette reading/cm^3			
Initial burette reading/cm^3			
Volume of FA5 use//cm^3			

Value used to calculate average =
……cm^3
Average volume of FA3 used =
……...cm^3

Questions:

a) Calculate the number of moles of sulphuric acid that:

(i) Did not react with the metal oxide, XO.

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(ii) Reacted with the metal oxide, XO.

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b) Determine the

(i) number of moles of the metal oxide, XO that reacted with sulphuric acid in FA3.

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(ii) molar mass of the metal oxide and hence the value of X in XO (O =16)

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Experiment 11:

You are provided with the following

FA1 which is a solution containing 1.18g of manganate (VII) ions in 500cm^3 of solution.

FA2 which is a solution of oxalic acid.

FA3 which is 1M sulphuric acid

Solid N which is an impure manganese (IV) oxide, MnO_2 referred to as pyrolusite.

Aim:

To determine the:

(i) molarity of manganate (VII) ions in FA1 in moles per litre.

(ii) Percentage impurity in the manganese (IV) oxide sample used. (Mn = 55, O =16)

Theory:

The oxalate ions from oxalic acid react with manganate (VII) ions and manganese (IV) oxide respectively according to the equations below.

$$2MnO_4^-{}_{(aq)} + 16H^+{}_{(aq)} + 5C_2O_4^{2-}{}_{(aq)} \longrightarrow 2Mn^{2+}{}_{(aq)} + 8H_2O_{(l)} + 10CO_{2(g)}$$

$$MnO_{2(s)} + 4H^+{}_{(aq)} + + C_2O_4^{2-}{}_{(aq)} \longrightarrow Mn^{2+}{}_{(aq)} + 2H_2O_{(l)} + 2CO_{2(g)}$$

PART I Procedure:

Transfer 50cm^3 of FA2 into a clean beaker using a measuring cylinder. Add 75cm^3 of distilled water. Label this solution FA4

Pipette 20 or 25cm^3 of FA4 into a clean conical flask. Add an equal volume of FA3 and heat the solution mixture to a temperature of about 70^{o}C and immediately titrate the hot solution mixture with FA1 from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your findings in the table 1 below:

Volume of pipette used ……...cm^3

Table I range:

25.00cm^3 to 25.30cm^3 if 25cm^3 is used.

20.00cm^3 to 20.30cm^3 if 20cm^3 is used.

Final burette reading/cm^3			
Initial burette reading/cm^3			
Volume of FA3 use//cm^3			

Value used to calculate average =

……cm^3

Average volume of FA3 used =

……...cm^3

Questions:

a) Calculate the molarity of MnO_4^- in FA1

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b) Determine the molarity of $C_2O_4^{2-}$ in FA2

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Procedure II

(i) Weigh accurately 1.0g of N and transfer it into a clean conical flask. By use of a measuring cylinder, transfer 150cm^3 of **FA2** followed by 50cm^3 of **FA3** into the flask containing solid N.

(ii) Transfer the mixture and boil gently for about 5 to 6 minutes (until the remaining solid particles turn brown). Cool the mixture and transfer in into a 250cm^3 volumetric flask and make it up to the mark with distilled water. label the solution **FA5**.

(iii) Measure and transfer 50cm^3 of **FA1** into a clean beaker. Add 50cm^3 distilled water and label this solution **FA6**.

(iv) Pipette 20 or 25cm^3 of **FA5** into a clean conical flask. Add an equal volume of **FA3** using a measuring cylinder and heat the mixture to about 70^{o}C and immediately titrate the hot solution with **FA6** from the burette until the end point is reached. Repeat the titration until you obtain consistent results. Record your results in the table below.

Mass of beaker + N ……...g
Mass of beaker a lone……...…...g
Mass of N ……...…...g
Volume of pipette used ……...cm^3

Table II range:
16.80cm^3 to 17.00cm^3 if 25cm^3 is used.
13.50cm^3 to 13.70cm^3 if 20cm^3 is used.

Final burette reading/cm^3			
Initial burette reading/cm^3			
Volume of **FA6** use//cm^3			

Value used to calculate average =
……cm^3
Average volume of FA6 used =
……...cm^3

Questions:

a) Calculate the number of moles of:

(i) MnO_4^- in FA6 that reacted with $C_2O_4^{2-}$ in FA5.

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(ii) $C_2O_4^{2-}$ in FA5 that reacted with MnO_4^- in FA6.

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(iii) $C_2O_4^{2-}$ in FA2 that reacted with the MnO_2.

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(iv) MnO_2 that reacted with the $C_2O_4^{2-}$ in FA2

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b) Determine the percentage impurity of manganese (IV) oxide in the sample used.

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Experiment 12:

You are provided with the following

FA1 which is Iodine solution.

FA2 which.is a solution containing 9.3g of hydrated sodium thiosulphate, $Na_2S_2O_3.5H_2O$ in $500cm^3$.

Solid W which is an impure sample of sodium sulphite, Na_2SO_3.

Aim: You are required to determine the:

(i) **Molarity of iodine in FA1**.

(ii) **Percentage impurity** of sodium sulphite in the sample.

Theory:

Sulphite ions formed are oxidized by iodine to sulphate ions according to the reaction below.

$$SO_3^{2-}{}_{(aq)} + H_2O_{(l)} + I_{2\,(aq)} \longrightarrow SO_4^{2-}{}_{(aq)} + 2I^-{}_{(aq)} + 2H^+{}_{(aq)} \quad ...(i)$$

Since the hydrogen ions produced by the above reaction is capable of reacting with thiosulphate ions, which would result into precipitation of sulphur, so some few solids of sodium hydrogen carbonate are added in the flask in order to remove all the hydrogen ions from the solution, before titration with standard sodium thiosulphate as shown below:

$$2HCO_3^-{}_{(aq)} + 2H^+{}_{(aq)} \longrightarrow 2CO_{2\,(g)} + 2H_2O_{(l)} \quad ....(ii)$$

The overall equations (i) and (ii) are as below:

$$SO_3^{2-}{}_{(aq)} + I_{2\,(aq)} + 2HCO_3^-{}_{(aq)} \longrightarrow SO_4^{2-}{}_{(aq)} + 2I^-{}_{(aq)} + 2CO_{2\,(g)} + 2H_2O_{(l)}$$

Procedure I

Pipette 20 or $25cm^3$ of FA1 in to a clean conical flask and titrate it with FA2 from the burette until the solution becomes pale yellow, then add 5 drops of starch indicator and continue with the titration until the blue – black starch – iodine complex just turns colourless. Repeat the titration until you obtain consistent results. Record your results in the table 1 below:

Table II range:

$10.50cm^3$ to $10.70cm^3$ if $25cm^3$ is used.

$8.50cm^3$ to $8.70cm^3$ if $20cm^3$ is used.

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Final burette reading/cm^3			
Initial burette reading/cm^3			
Volume of FA2 use//cm^3			

Value used to calculate average =

……cm^3

Average volume of FA2 used =

……...cm^3

Questions:

a) Write the equation for the between iodine and thiosulphate ions.

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b) Calculate the molarity of the:

(i) Sodium thiosulphate in FA2 in moles per dm^3

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(ii) Iodine in FA1 in moles per dm^3

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Procedure II

(i) Weigh accurately 1.0g of W into a clean beaker. Add 100cm^3 distilled water and stir well to dissolve. Transfer the solution into a 250cm^3 volumetric flask and make it up to mark with distilled water. label this solution **FA3**.

(ii) Using a measuring cylinder, measure and transfer 70cm^3 of **FA1** into a clean conical flask. Using another measuring cylinder, transfer 30cm^3 of **FA3** followed by 2.0g of sodium hydrogen carbonate, and continue shaking well to dissolve. Label the resultant solution **FA4**.

(iii) Pipette 20 or 25cm^3 of FA4 in to a clean conical flask and titrate it with FA2 from the burette until the solution becomes pale yellow, then add 5 drops of starch indicator and continue with the titration until the blue – black starch – iodine complex just turns colourless. Repeat the titration until you obtain consistent results. Record your results in the table 1 below

Table II range:

8.80cm^3 to 9.00cm^3 if 25cm^3 is used.

7.00cm^3 to 7.20cm^3 if 20cm^3 is used.

Final burette reading/cm^3			
Initial burette reading/cm^3			
Volume of FA2 use//cm^3			

Value used to calculate average =

……cm^3

Average volume of FA2 used =

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……...cm^3

Questions:

a) Calculate the number of moles of iodine in FA1 that reacted with sulphite ions in FA3.

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b) Determine the mass of pure sodium sulphite in the:

(i) $30cm^3$ of FA3 that reacted with iodine in FA1 (Na =23, S =32, O =16)

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(ii) $250cm^3$ of FA3 and hence the percentage impurity of sodium sulphite in the sample used in FA3.

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Experiment 13:

You provided with the following:

FA1 which is a solution containing manganate (VII) solution

FA2 which is a solution containing 2.64g of an impure metal persulphate (Peroxodisulphate), $\mathbf{M_2S_2O_8}$ in $200cm^3$ of solution.

FA3 which is a 2M sulphuric acid.

Solid X which is diammonium iron (II) sulphate hexahydrate, $(NH_4)_2SO_4FeSO_4.6H_2O$.

Aim:

You are required to determine the:

(i) Molar concentration of manganate (VII) ions in FA1.

(ii) Percentage purity of the sample of the metal persulphate, $\mathbf{M_2S_2O_8}$

Theory:

Persulphate ions react with excess iron (II) ions according to the equation below:

$\mathbf{S_2O_8^{2-}{}_{(aq)} + 2Fe^{2+}{}_{(aq)} \quad 2SO_4^{2-}{}_{(aq)} + 2Fe^{3+}{}_{(aq)}}$

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The unreacted iron (II) ions are then titrated with acidified manganate (VII) ions according to the equation below:

$MnO_4^-{}_{(aq)} + 8H^+{}_{(aq)} + 5Fe^{2+}{}_{(aq)} \longrightarrow Mn^{2+}{}_{(aq)} + 5Fe^{3+}{}_{(aq)} + 4H_2O_{(l)}$

Procedure I

Weigh accurately 6.3g of solid X and dissolve in about 100cm^3 of distilled water in a beaker. Transfer the dissolved solution into a 250cm^3 volumetric flask and make it up to the mark with distilled water. Label this solution **FA4**. Pipette 20 or 25cm^3 of **FA4** in to a clean conical flask. Add an equal volume of **FA3** and titrate it with **FA1** from the burette until the solution becomes pale yellow, then add 5 drops of starch indicator and continue with the titration until the blue – black starch – iodine complex just turns colourless. Repeat the titration until you obtain consistent results. Record your results in the table 1 below

Table I range:

21.40cm^3 to 21.60cm^3 if 25cm^3 is used.

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

Value used to calculate average =

……cm^3

Average volume of FA1 used =

……..cm^3

Questions:

a) Determine the molar concentration of:

(i) Fe^{2+} ions in FA4.

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(ii) Manganate (VII) ions in FA1

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Procedure II

(i) Using a measuring cylinder, obtain 15cm^3 of **FA2** and transfer it into a clean beaker followed by 85cm^3 of **FA4**. Shake well and label the solution **FA5**.

(ii) Pipette 20 or 25cm^3 of **FA5** in to a clean conical flask. Add an equal volume of **FA3** and titrate it with **FA1** from the burette until the solution becomes pale yellow, then add 5 drops of starch indicator and continue with the titration until the blue – black starch – iodine complex just turns colourless. Repeat the titration until

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you obtain consistent results. Record your results in the table 2 below

Table II range:

25.50cm^3 to 25.70cm^3 if 25cm^3 is used.

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

Value used to calculate average =

……cm^3

Average volume of FA1 used =

……...cm^3

Questions:

a) Calculate the number of moles of:

(i) Manganate (VII) ions that reacted with the excess Fe^{2+} ions in FA5

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(ii) Fe^{2+} ions in FA4 that did not react with persulphate ions (Peroxodisulphate ions) in FA2.

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(iii) Fe^{2+} ions in FA4 that reacted with persulphate ions (Peroxodisulphate ions) in FA2.

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(iv) Persulphate ions in 200cm^3 of FA2.

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b) Determine the mass of the metal persulphate,), **$M_2S_2O_8$** in 200cm^3 of FA2 and hence calculate the percentage purity of the metal persulphates. (M =39, S =32 O = 16)

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Remember to revise also the following areas:

1. Chemical Energetics (Heat changes)
2. Chemical Kinetics (Rates of reactions)
3. Partition coefficient (KD)

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