Laboratory Task 1: Organic Synthesis

At the work bench

For preparations of compounds

▪ Beakers	3
▪ Dropper (plastic)	1
▪ Filter paper (circles)	2
▪ Funnels	2
▪ Glass rod	2
▪ Measuring cylinder (10mL)	1
▪ Test tube rack	1
▪ Wash Bottle	1
▪ Chemicals	10% NaOH, 15 mL
	1:1 HCl, 15 mL
	$NaNO_2$, 5 mL
	pH paper
▪ Vials	p-nitroaniline, 0.6 g/vial (2 vials)
	Compound **B** and **C** (1vial each)

For qualitative tests

▪ Droppers	2
▪ Cavity plate	1
▪ Chemicals	Compound **B** and **C**
	Ethanol, 2 mL
	Aq. $FeCl_3$
	NaOH
	Sat. $NaHCO_3$
	2,4-DNP

Preparation of Azo dyes (40 Marks)

Azo dyes are an important class of dyes. They are colored due to the presence of –N=N- group. In their preparation, aromatic primary amines are diazotised using sodium nitrite and an acid, like HCl (see eq.1). The diazonium salts are coupled with active aromatic compounds to obtain azo dyes (see eq.2).

$Ar\text{-}NH_2 + NaNO_2 + 2HCl \rightarrow Ar\text{-}N_2^+Cl^- + NaCl + 2H_2O$ …………1

$Ar\text{-}N_2^+ Cl^- + Ar'H \rightarrow Ar\text{-}N{=}N\text{-}Ar' + HCl$ ………… 2

Identification of functional groups in compounds B and C

You are given two compounds Compound **B** (mol.wt.138) and **C** (mol.wt.144) (**Kept in plastic storage vials)**. Compounds **B** and **C** contain C, H and O. You will be using these compounds for coupling with a diazonium salt to prepare the azo dyes I and II.

Conduct appropriate tests with the reagents provided to you and identify the functional groups present in **B** and **C**. Write your observations in the following table.

Test	**Observation**	**Functional group present**
Pinch of Comp B + Sat. $NaHCO_3$		
Pinch of Comp B +10% aq. NaOH		
Pinch of Comp B + few drops of alcohol + Aq. $FeCl_3$		
Pinch of Comp B + few drops of alcohol + 2,4-DNP		

(3 marks)

Test	**Observation**	**Functional group present**
Pinch of Comp C + Sat. $NaHCO_3$		
Pinch of Comp C +10% aq. NaOH		
Pinch of Comp C + few drops of alcohol + Aq. $FeCl_3$		
Pinch of Comp C + few drops of alcohol + 2,4-DNP		

(3 marks)

Preparation of Azo dye I

Step I: Diazotization of 4-nitroaniline

Empty one vial of 4-nitroaniline (0.600g) in a beaker. Add 4 mL of supplied HCl solution and stir the solution. Cool the solution in an ice bath to 0-5°C with stirring. A solid may appear at this stage. Simultaneously cool the $NaNO_2$ solution provided to you. After both the solutions are properly cooled, add 2 mL of the $NaNO_2$ solution to the other solution, slowly with stirring.

Step II: Coupling

Dissolve the given quantity of Compound **B** in 8 mL of 10% NaOH solution in a beaker. Cool the solution in an ice bath to about 0-5°C. Slowly add the solution obtained in **step I** with stirring. A coloured compound separates. Stir for about 5 minutes. At this stage, add about 1.5mL of HCl solution and stir the solution. Check the pH with pH paper (about 3). If needed, add more HCl with the help of a dropper to reach the desired pH. Stir the solution and keep it in ice bath for 15 minutes. Filter the solution. Use 50 ml of distilled water for transferring and washing the precipitate.

Preparation of Azo dye II

Step I : Diazotization of 4-nitroaniline

Empty one vial of 4-nitroaniline (0.600g) in a beaker. Add 4 mL of supplied HCl solution and stir the solution. Cool the solution in an ice bath to 0-5°C with stirring. A solid may appear at this stage. Simultaneously cool the $NaNO_2$ solution provided to you. After both the solutions are properly cooled, add 2 mL of the $NaNO_2$ solution to the other solution, slowly with stirring.

Step II : Coupling

Dissolve the given quantity of Compound **C** in 6 mL of 10% NaOH solution in a beaker. Cool the solution in an ice bath to about 0-5°C. Slowly add the solution obtained in **step I** with stirring. Stir for about 5 minutes. At this stage, add about 1.00 mL of HCl with stirring. Check the pH with pH paper (about 3). If needed, add more HCl with the help of a dropper to reach the desired pH. Stir the solution and keep it in ice bath for 15 minutes. Filter the solution. Use 50 ml of distilled water for transferring and washing the precipitate.

Laboratory Task 1 — Answersheet

1.1 Quantity of 4-nitroaniline taken for preparation of each dye = 0.600g

The coupling reactions with compound **B** and **C** are equimolar. Calculate the quantities of **B** and **C** required for the reactions.

Amount of **B:** [g]

(1 mark)

Amount of **C:** [g]

(1 mark)

1.2 **Azo Dye I**

(a) Colour of azo dye **I** [ ]

(3 marks)

(b) The mass of azo dye **I** [g]

(6 marks)

(c) Theoretical yield on the basis of mass of 4-nitroaniline [g]

(1 mark)

(d) The yield obtained as a percentage of the theoretical yield:

[%]

(1 mark)

1.3 **Azo Dye II**

(a) Colour of azo dye **II**

(3 marks)

(b) The mass of azo dye **II** g

(6 marks)

(c) Theoretical yield on the basis of the mass of 4-nitroaniline g

(1 mark)

(d) The yield obtained as a percentage of theoretical yield. %

(1 mark)

1.4 Write the equation for diazotization of 4-nitroaniline.

(1 mark)

1.5 Why is the coupling reaction involved in the experiment carried out in an alkaline medium?

(1 mark)

1.6 Why the solutions were made acidic before filtration of the final products?

(1 mark)

1.7 If, in the azo coupling reaction, the diazotized 4-nitroaniline is coupled with 4-methyl phenol, write the balanced equation of the azo coupling reaction.

(1 mark)

Procedure for TLC

For Azo Dye I

Dissolve a pinch of azo dye I in a small quantity of acetone in a sodium fusion tube. Obtain a TLC plate from the laboratory expert. Draw a faint line, at a distance of about 1cm from the edge of the plate. Using a thin capillary tube, place a drop of azo dye **I** on the line drawn on the plate. In a similar manner, spot azo dye **II**. Allow the spots to dry. Then place the plate in a beaker, containing methanol. Cover the beaker with a watch glass, and allow the solvent to rise appreciably (approximately 1 cm away from the top). Remove the plate from the beaker and mark the solvent front immediately. Calculate the R_f values using the formula given below and record the result in the answer sheet.

$$R_f = \frac{\text{distance travelled by the compound}}{\text{distance travelled by the solvent front}}$$

Submit your TLC plates to the expert before leaving the laboratory.

The R_f values for azo dyes I and II

a) R_f for azo dye I:

(3 marks)

b) R_f for azo dye II:

(3 marks)

Laboratory Task 2: Analysis of an inorganic complex

At the work bench

▪ Burette 50 mL	1
▪ Conical flasks	2
▪ Funnel	1
▪ Filter paper	2
▪ Measuring cylinder 50mL	1
▪ Wash Bottle	1
▪ Chemicals	$KMnO_4$, 70 mL
	H_2SO_4 4 M, 60 mL
	Zn dust, 2.5g/vial (2 vials)
	Sample, 0.25g/vial (2 vials)
▪ For qualitative tests	NaOH 2 M
	KSCN 0.1 M
	HCl 0.1 M
	Solution **1**, [Fe(NO)$_3$]
	Solution **2** (This is the solution of the complex)

(molarity of $KMnO_4$ will be supplied to you)

Laboratory Task 2 **(40 marks)**

Analysis of an inorganic complex

You are given an inorganic complex containing iron, oxalate, potassium and water. In part A of this experiment, you will be analyzing the given complex for its iron and oxalate contents. In part B, you will be performing some qualitative tests with solutions of complex and iron (III) nitrate. The qualitative tests are conducted for comparing the strength of different ligands that bind with iron.

PART A

Determination of the oxalate content

A sample in duplicate is given to you for analysis. The mass of the sample is stated on the vial.

1. Transfer the content of one vial completely to a clean conical flask.
2. Add 25 mL of 4 M H_2SO_4. Heat the solution on a hot plate to 70-80^{0}C.
3. Remove the flask from the hot plate (use gloves to hold the hot flask) and titrate the hot solution against $KMnO_4$ till it is light pink in colour.

   Do not discard the content after the titration, as you will be estimating the iron from the same solution.

Determination of the iron content

1. After the titration of the oxalate, to the same solution, carefully add one vial of zinc powder provided to you.
2. After 1 or 2 minutes, keep the solution on the hot plate. Boil the solution for 10-15 minutes.
3. Carefully remove the flask from the hot plate (use the gloves) and allow the solution to cool.
4. If necessary, filter the solution using a filter paper.
5. Titrate the solution/filtrate against supplied $KMnO_4$ solution.
6. Perform both the titrations with another sample provided to you. Enter your results in the answer sheet.

PART B

Comparing strength of different ligands

You are given 0.1 M solution of $Fe(NO_3)_3$ (that is, Solution 1**) and of the complex (that is,** Solution 2**). In the** solution I**, iron exists as $[Fe(H_2O)_6]^{3+}$.**

Carry out the following tests and report your observations.

Solution tested 5 drops	Reagent to be added - 5 drops	Observations
Solution 1	2 M NaOH	
Solution 2	2 M NaOH	
Solution 1	0.1 M HCl	
Solution 2	0.1 M HCl	
Solution 1	0.1 M KSCN	
Solution 2	0.1 M KSCN	

(3 marks)

Based on the observations, arrange the ligands, that is, H_2O, OH^-, Cl^-, SCN^- and $C_2O_4^{-2}$ on the basis of their binding strength with iron. Explain your answer in brief.

(4 marks)

Laboratory Task 2 **Answer Sheet**

Concentration of $KMnO_4$: ____________________ M

2.1

	Trial I		Trial II	
	Titration 1	**Titration 2**	**Titration 1**	**Titration 2**
Initial burette reading (mL)				
Final burette reading (mL)				
Volume of $KMnO_4$ (mL)				

2.2 Write the balanced chemical equation for the reaction involved in the titration of oxalate with $KMnO_4$. State why the titration is carried in an acidic medium and in hot condition.

(2 marks)

2.3 State what happens

a) immediately after addition of Zn dust to the flask and

b) when the solution is boiled.

(1 mark)

2.4 Why is Zn dust added after titration of oxalate is complete?

(1 mark)

2.5 If any Zn dust remains in solution after boiling, it is necessary to remove it by filtration, before titrating iron with $KMnO_4$. Explain why?

(1 mark)

2.6 Write the balanced chemical equation for the reaction involved in the titration of iron.

(1 mark)

2.7 Calculate the amount of iron and oxalate for any one of the trials. (Show the main steps in your calculation).

(22 marks)

2.8 Also, calculate the potassium and water content of the complex.

(4 marks)

2.9 The molar ratio of iron: oxalate: potassium: water in the given complex is

(1 mark)

Laboratory Task 3: Distribution coefficient of benzoic acid

At the work bench

▪ Burette 25mL	1
▪ Conical flasks 100mL	4
▪ Pipette (2mL and 5mL)	2
▪ Funnel	1
▪ Plastic bottles	4
▪ Wash Bottle	1
▪ Water bath	1
▪ Chemicals	For aq. Layer NaOH, 50 mL For org layer NaOH, 80 mL

(molarities of the NaOH solutions will be supplied to you)

Laboratory Task 3 (40 marks)

Study the association of benzoic acid from its distribution between toluene and water

In this experiment, you will be studying the association of benzoic acid based on its distribution between toluene and distilled water. After the equilibrium is reached, the quantity of benzoic acid in both layers (organic and aqueous) is estimated by titrating the layers with standardized solutions of alkali.

For the distribution of a weak organic acid between an organic solvent and water, the distribution coefficient (K_D) is given as $K_D = \frac{(1-\alpha_o)C_o}{(1-\alpha_w)C_w}$ ……….1

α_o = fraction of solute associated in organic solvent

α_w = fraction of solute dissociated in aqueous solvent

C_w = Concentration of benzoic acid in Aqueous layer

C_o = Concentration of benzoic acid in Organic layer

If the solute (represented as B in equation) undergoes association in organic solvent, that is, $nB \rightleftharpoons B_n$ and has a normal molecular mass in water, the association constant K is given by

$$K = \frac{\alpha_o C_o}{n(1-\alpha_o)^n C_o^n} \qquad \text{…………2}$$

Using 1 and 2 and $\alpha_w \approx 0$, gives $K_D = \frac{(\alpha_o C_o / nK)^{1/n}}{C_w}$

If $\alpha_o \approx 1$ then $\frac{C_o^{1/n}}{C_w}$ = constant

Procedure

<u>Preparation of mixtures</u>

You are given the following stock solutions

1. Benzoic acid in toluene 2.Toluene 3.Distilled water

Prepare the mixtures as given in the table below. The required quantities can be taken using burettes arranged at one place. Laboratory expert will call each one of you. Take the plastic bottles kept on your table and obtain the stated quantities of solutions in each bottle.

Solution	**Bottle I** mL	**Bottle II** mL	**Bottle III** mL	**Bottle IV** mL
Benzoic acid in toluene (1)	20.0	17.5	15.0	12.5
Toluene (2)	5.0	7.5	10.0	12.5
Distilled water (3)	25.0	25.0	25.0	25.0

The distilled water, which is to be added to each bottle is kept on your table. Use the burette kept on your table to take the stated volume of distilled water. Stopper the bottles and shake the bottles in rotation for 20 minutes. Place all the bottles in water bath and allow them to attain the equilibrium for 10 minutes.

Estimation of Benzoic acid

Titration of the Aqueous layer

1. Fill the burette with NaOH solution provided to you. *(Concentration of NaOH will be supplied to you)*
2. Using a pipette, pipette out 5 mL of the aqueous layer, from **Bottle IV** into a conical flask. Titrate this aqueous layer against NaOH using phenolphthalein as an indicator.
3. Repeat the same procedure for aqueous layers from **Bottle III, II** and **I** respectively. *(Perform the titrations in the given order)*
4. Enter your results in the answer sheet.

Titration of the Organic layer

1. Fill the burette with NaOH solution provided to you. *(Concentration of NaOH will be supplied to you)*
2. Using a pipette, pipette out 2 mL of the organic layer, from **Bottle IV** into a conical flask. Add 5 mL of distilled water to the flask.
3. Titrate the organic layer against NaOH using phenolphthalein as an indicator.
4. Repeat the same procedure for organic layers from **Bottle III, II** and **I** respectively. *(Perform the titrations in the given order)*
5. Enter your results in the answer sheet.

Laboratory Task 3 Answersheet

Concentration of NaOH for aqueous layer: ____________________ M

Concentration of NaOH for organic layer: ____________________ M

Titration Readings	**Bottle I**	**Bottle II**	**Bottle III**	**Bottle IV**
For Aqueous Layer (mL)				
For Organic Layer (mL)				

3.1 Write a balanced chemical equation for the reaction involved in the titration.

(0.5 mark)

3.2 For any one bottle, show the calculation for concentration of benzoic acid in each layer.

(1 mark)

3.3 Concentration in (M) of benzoic acid in both the layers:

Titration Readings	**Concentration of benzoic acid in Aqueous layer (C_w)**	**Concentration of benzoic acid in Organic layer (C_o)**	**Log (C_w)**	**Log (C_o)**
Bottle I				
Bottle II				
Bottle III				
Bottle IV				

(24 marks)

3.4 Plot a graph of log (C_w) vs log (C_o) and calculate the association number (n) using equation $\log C_w = \frac{1}{n}\log C_o + \text{cons tant}$. Hence determine $\frac{C_o^{1/n}}{C_w}$.

(8 marks)

3.5 With the calculated **n** value, comment on the state of benzoic acid in the organic layer.

(0.5 mark)

3.6 The structure of isoamyl alcohol is as follows:

$$H_3C\underset{\displaystyle CH_3}{\underset{|}{C}}HCH_2CH_2OH$$

If the same experiment is carried out using iso-amyl alcohol and distilled water, the observed n value will be **(Mark X in the correct Box)**

a) Same as that for toluene ☐

b) Less than that for toluene ☐

c) More than that for toluene ☐

Justify your answer

(2 marks)

1.7 If the experiment done by you is repeated at 50°C, which of the following statements will be true? **(Mark X in the correct Box).**

a) Both the partition coefficient and value of n will change. ☐

b) The change in temperature will not have any effect on the partition coefficient and the value of n . ☐

c) The partition coefficient will change, but value of n will not. ☐

d) The value of n will change, but partition coefficient will not. ☐

(1 mark)

1.8 When $\alpha_w \approx 0$ and $\alpha_o \approx 1$ then $\frac{C_o^{1/n}}{C_w} = K_D$. Derive an expression for dissociation constant of benzoic acid (K_a) in terms of C_o, C_w and K_D.

(3 marks)

Laboratory Task 1: Inorganic Estimation

At your work bench

▪ Burette 25 mL	2
▪ Conical flasks (containing sample)	2
▪ Funnel	2
▪ Measuring cylinder 10mL	1
▪ Wash Bottle	1
▪ Chemicals	Na_2EDTA, 70 mL
	$Pb(NO_3)_2$, 70 mL
	Hexamine, 4.5g/vial (2 vials)
	Xylenol Orange indicator

(molarities of Na_2EDTA, $Pb(NO_3)_2$, will be supplied to you)

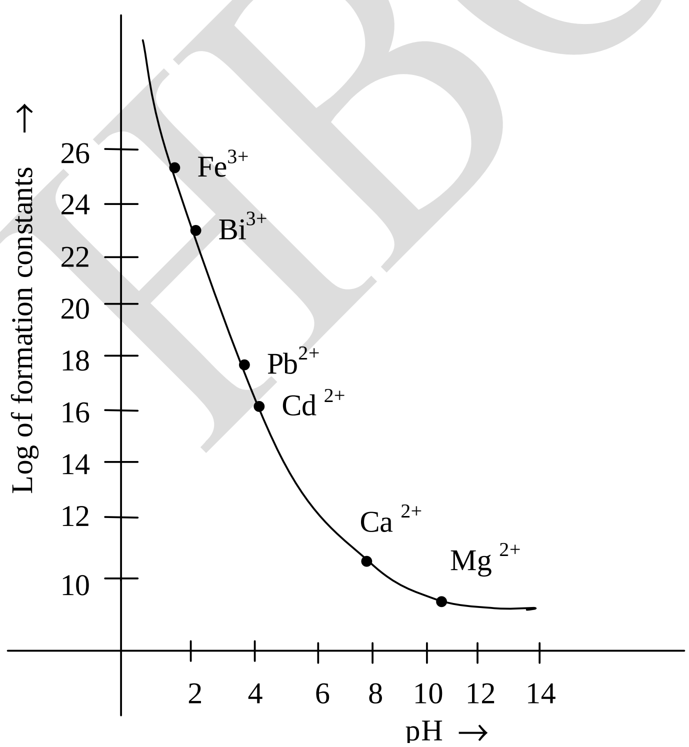
Laboratory Task 1 (33 marks)

To estimate the amounts of lead(II), bismuth(III) and cadmium(II) in a given sample by complexometric titration

In this experiment, you will be estimating lead(II), bismuth(III) and cadmium(II) from the given sample solution. The titrant used is disodium salt of Ethylene diamine tetraacetic acid (abbreviation: Na_2EDTA or Na_2H_2Y). Regardless of the charge on the metal ion, the titrant forms stable 1:1 complex with the metal ion.

The indicators used in the complexometric titrations are called as the metal-ion indicators that form stable complexes with the metal ions. The colour of the free indicator and that of the indictor metal ion complex differ from each other. The metal-ion indicators themselves are either weak acids or weak bases. The indicator used in the present experiment is xylenol orange, a weak acid.

The graph given below is the graph of log of the formation constants of the complexes of the metal ions with EDTA, as a function of pH. The metal ions you will be estimating are indicated on the graph.



You are supplied with two burettes (25 mL). You are expected to fill one burette with Na_2EDTA solution and the other with the standard Pb (II) solution. The sample solution is supplied to you in duplicate. If you do not get consistent readings for the two trials then only ask for a third sample.

Titration 1

Note that the pH of the sample solution is between 1-2. To the sample solution, add 4 to 5 drops of xylenol orange indicator and shake the contents of the flask. Titrate the solution against the standard Na_2EDTA solution until the colour changes from red to orange yellow. Record your burette reading (**A** mL).

Titration 2

To the same flask, add one vial of solid hexamine supplied to you. Shake the contents well for a minute. At this point, the colour of the solution will change to red. Titrate the solution against the standard Na_2EDTA solution till the colour changes to yellow. Record your burette reading (**B** mL).

Titration 3

To the same flask, add 10 mL of 1,10-phenanthroline solution, using a measuring cylinder. This reagent selectively reacts with Cd and forms a stable Cd-phenanthroline complex. This complex is more stable than Cd-EDTA complex. Shake the contents of the flask thoroughly and titrate against the standard Pb(II) solution till the colour changes to reddish orange. Record the burette reading (**C** mL).

Repeat the above procedure for Trial II.

Laboratory Task 1 Answersheet

Concentration of standard Na_2EDTA : ________________

Concentration of Pb (II) solution : ________________

	Trial I			**Trial II**		
	Titration 1(A mL)	**Titration 2(B mL)**	**Titration 3(C mL)**	**Titration 1(A mL)**	**Titration 2(B mL)**	**Titration 3(C mL)**
Initial burette reading						
Final burette reading						
Volume of Na_2EDTA						

(22 marks)

1.1 Use the graph and find out the metal ion that will be titrated at the original pH of the solution.

(0.5 mark)

1.2 Write the balanced chemical equation for the reaction involved in Titration 1 (use abbreviation Na_2H_2Y for EDTA)

(1 mark)

1.3 From the graph, give the value of the formation constant of the metal -EDTA complex formed in **Titration 1**.

(0.5 mark)

1.4 State the purpose of addition of hexamine before the **Titration 2**

(1 mark)

1.5 After the addition of hexamine, why does the colour of the solution change from yellow to red?

(1 mark)

1.6 The colour change at the end point (red to orange yellow) in the **Titration 1** is due to

(a) the formation of the metal-indicator complex. ☐

(b) the release of the free indicator from the metal-indicator complex. ☐

(c) the formation of metal-EDTA complex. ☐

[Mark **X** in the correct box.] **(1mark)**

1.7 Calculate the amount of Bi(III), Pb (II) and Cd (II) in gram for any **one** of the two/three trials. (Show the main steps in your calculation.)

(6 marks)

Amount of Bi(III) in given sample	g
Amount of Pb(II) in given sample	g
Amount of Cd(II) in given sample	g

Laboratory Task 2: Inorganic Preparation, Estimation and qualitative tests

At your work bench

Preparation (Part A)

▪ Beakers 100 mL	2
▪ Glass rod	1
▪ Ice Bath	1
▪ Measuring cylinder 10mL	1
▪ Chemicals	$Fe(NO_3)_3.9H_2O$, 3.000g/vial (1 vial)
	$K_2C_2O_4$, (1 vial)

Estimation (Part B)

▪ Burette 50 mL	1
▪ Conical flasks	2
▪ Funnel	1
▪ Filter paper	2
▪ Measuring cylinder 50mL	1
▪ Wash Bottle	1
▪ Chemicals	$KMnO_4$, 70 mL
	H_2SO_4 4 M, 60 mL
	Zn dust, 2.5g/vial (2 vials)

Qualitative Tests (Part C)

▪ Cavity Plate	1
▪ Droppers	4
▪ Chemicals	NaOH 2 M
	KSCN 0.1 M
	HCl 0.1 M
	Solution **1**, [$Fe(NO_3)_3$]
	Solution **2** (This is the solution of the complex)

(molarity of $KMnO_4$ will be supplied to you)

Laboratory Task 2 (40 marks)

Synthesis and analysis of an inorganic complex

In this experiment you will prepare a co-ordination complex of Fe (III) and oxalate in part A. In part B of the experiment, you will be analyzing the given complex for its iron and oxalate contents using titration technique. In part C, you will be performing some qualitative tests with solutions of the complex and iron (III) nitrate. The qualitative tests are conducted for comparing the strength of different ligands that bind with iron.

PART A: Synthesis of the complex

You are supplied with 3.000 g of $Fe(NO_3)_3.9H_2O$ in a vial and potassium oxalate in another vial. Transfer the Fe(III) nitrate in a beaker and dissolve it in 3 mL of water. In case of any precipitate, filter the solution. In another beaker, dissolve the potassium oxalate in 8 mL of hot water. Add the Fe(III) nitrate solution slowly, to the oxalate solution with constant stirring. After the entire addition is done, heat the contents for 5 minutes on the hot plate. Remove the beaker from hot plate and allow it to cool for 2 minutes. Then transfer the beaker to ice bath and keep it in ice bath for 15 to 20 minutes. The laboratory expert will collect your beaker for filtration and the product will be given back to you on your table. Allow the product to dry for 15 to 20 minutes. At the end of this time interval, carefully transfer the product on the pre-weighed butter paper supplied to you. Take the product for weighing to the laboratory expert.

PART B

<u>Determination of the oxalate content</u>

A sample of 0.250 g will be weighed in duplicate for analysis.

1. Transfer the contents of one vial completely to a clean conical flask.
2. Add 25 mL of 4 M H_2SO_4. Heat the solution on a hot plate to 70-80^{0}C.
3. Remove the flask from the hot plate (use gloves to hold the hot flask) and titrate the hot solution against $KMnO_4$ till it is light pink in colour.

Do not discard the contents after the titration, as you will be estimating iron from the same solution.

Determination of the iron content

1. After the titration of the oxalate, to the same solution, carefully add one vial of zinc powder provided to you.
2. After 1 or 2 minutes, keep the solution on the hot plate. Boil the solution for 10-15 minutes.
3. Carefully remove the flask from the hot plate (use the gloves) and allow the solution to cool.
4. If necessary, filter the solution using a filter paper.
5. Titrate the solution/filtrate against supplied $KMnO_4$ solution.
6. Perform both the titrations with another sample provided to you. Enter your results in the answer sheet.

Laboratory Task 2 **Answersheet**

Concentration of $KMnO_4$: ______________ M

2.1

	Trial I		Trial II	
	Titration 1	**Titration 2**	**Titration 1**	**Titration 2**
Initial burette reading (mL)				
Final burette reading (mL)				
Volume of $KMnO_4$ (mL)				

(16 marks)

2.2 After addition of 4 M H_2SO_4 the pH of the solution is close to 1. For oxalic acid, $pKa_1 = 1.27$ and $pKa_2 = 4.27$. When oxalate is released from the complex at this pH it will be converted to

$H_2C_2O_4$ ☐ $HC_2O_4^-$ ☐ $C_2O_4^{-2}$ ☐

(0.5 mark)

2.3 Write the balanced chemical equation for the reaction involved in the titration of oxalate with $KMnO_4$.

(0.5 mark)

2.4 State

i) Why is an acidic medium needed for the titration and

ii) Why is it necessary to carry out the titration in hot condition..

(1 mark)

2.2 Write the reactions that take place

a) **immediately** after addition of Zn dust to the flask and

b) when the solution is boiled.

(1 mark)

2.3 Why is Zn dust added **only after** the titration of oxalate is complete?

(0.5 mark)

2.5 If any Zn dust remains in solution after boiling, it is necessary to remove it by filtration, before titrating iron with $KMnO_4$. Explain why?

(0.5 mark)

2.6 Write the balanced chemical equation for the reaction involved in the titration of iron.

(1 mark)

2.7 Calculate the amount of iron and oxalate for any one of the trials. (Show the main steps in your calculation).

(2 marks)

2.8 Also, calculate the potassium and water content of the complex.

(3marks)

2.9 The molar ratio of iron: oxalate: potassium: water in the given complex is

2.10 Using the molar ratio of iron and oxalate calculated by you, write the balanced chemical equation/s for reaction/s involved in formation of the complex.

(1 mark)

2.11 Also calculate the theoretical yield of the complex from the mass of the Iron (III) nitrate supplied to you

(1 mark)

2.12 (a) Mass of the product g

(1 mark)

(b) The yield obtained as a percentage of the theoretical yield

%

(5 marks)

(1 mark for calculation and 4 marks for yield)

(c) Colour of the product

(1 marks)

PART C

Comparison of binding strength of different ligands

You are given 0.1 M solution of $Fe(NO_3)_3$ (that is, Solution 1**) and of the complex (that is,** Solution 2**). In the** solution I**, iron exists as $[Fe(H_2O)_6]^{3+}$.**

Carry out the following tests and report your observations.

Solution tested **(5 drops)**	**Reagent added – (5 drops)**	**Observations**
Solution 1	2 M NaOH	
Solution 2	2 M NaOH	
Soluton1	0.1 M HCl	
Solution 2	0.1 M HCl	
Solution 1	0.1 M KSCN	
Solution 2	0.1 M KSCN	

(3 marks)

Based on the observations, arrange the ligands, that is, H_2O, OH^-, Cl^-, SCN^- and $C_2O_4^{-2}$ on the basis of their binding strength with iron. Explain your answer in brief.

(2 marks)

Laboratory Task 3: Organic synthesis

At your work bench

Part I: Identification of functional groups in compounds A and B

▪ Droppers	4
▪ Cavity plate	1
▪ Chemicals	Compound **A** and **B**
	Sat. $NaHCO_3$,
	10% aq. NaOH,
	neutral $FeCl_3$ solution,
	Aq. $KMnO_4$,
	ammonical solution of $AgNO_3$.

Part II: Separation of the Given Mixture of Compounds A and B

▪ Beakers	2
▪ Dropper (plastic)	2
▪ Filter paper (circles)	1
▪ Funnel	1
▪ Glass rod	1
▪ Measuring cylinder (10mL)	2
(25 mL)	1
▪ Syringe (2mL)	1
▪ Wash Bottle	1
▪ Chemicals	Sat $NaHCO_3$, 30 mL
	1:1 HCl, 15 mL
	pH paper
	Mixture of Compound **A** and **B**

Part III: Preparation of Derivative

▪ Conical Flask	2
▪ Cork	1
▪ Filter paper (circle)	1
▪ Funnel	1
▪ Glass rod	1
▪ Ice bath	1
▪ Measuring cylinder (50 mL)	1
(10mL)	1
▪ Syringe (1mL)	1
▪ Wash Bottle	1
▪ Chemicals	Acetone, 5 mL Ethanol, 30 mL NaOH, 35 mL

Part IV: TLC

▪ Beaker	1
▪ Nesler Tubes	2
▪ Capillary	2
▪ TLC plate	1
▪ Watch glass	1
▪ Chemicals	Pet Ether and Choloroform mixture (90:10) (solvent system for TLC) acetone (solvent for dissolution of sample)

Laboratory Task 3 (40 Marks)

Identification of functional groups and synthesis of derivative

You are expected to complete Part I of this task in first 30 minutes and return the paper to the lab expert. At the end of this part, you will be given the paper for part II.

Part I: Identification of functional groups in compounds A and B

You are given two compounds Compound **A** (molecular mass 122) and Compound **B** (molecular mass 106). Following reagents are given to you: Sat. $NaHCO_3$, 10% aq. NaOH, neutral $FeCl_3$ solution, aq. $KMnO_4$, ammonical solution of $AgNO_3$.

Conduct appropriate tests with the reagents provided to you and identify the functional groups present in compound **A** and **B**. Write your observations in the following table.

Compound A

Test	**Observation**	**Functional group present**

(3 marks)

Compound B

Test	Observation	Functional group present

(3 marks)

From your conclusions regarding the functional groups present in compound **A** and **B**, suggest a scheme for their separation from their mixture. (hint: one of the reagents supplied can be used for the separation).

(2 marks)

Part II : Separation of the Given Mixture of Compounds A and B

A mixture of compounds A and B is supplied to you in a 100 mL beaker. Add 20 mL of saturated NaHCO3 solution to this mixture and stir the solution for 5 minutes (till effervescence ceases). Carefully, transfer the contents of the beaker to 25 mL measuring cylinder supplied to you. Use another 5 mL of saturated NaHCO3 to transfer the content completely. Allow the two layers to separate clearly. Remove the organic layer, with the help of a syringe and transfer it to a clean 10 mL measuring cylinder. Note the volume of the organic layer (compound B). You will be using this layer for the preparation of new compound (see Part III).

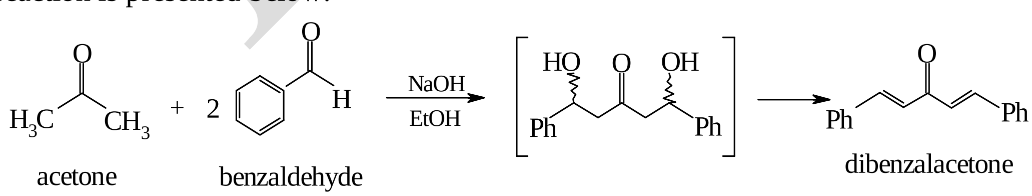
Volume of the organic layer (compound B) | |

Regeneration of compound A from the aqueous extract

Transfer the aqueous layer from the measuring cylinder to a 100 mL beaker. Add 50% HCl solution in small lots to the beaker with stirring till the solution is distinctly acidic (use pH paper). Note the volume of HCl added. Filter the precipitate (compound A) through filter paper. Use minimum amount of water for transferring and washing of the precipitate. The expert will collect your compound for drying.

Volume of acid needed for regeneration of compound A | |

The organic layer obtained in part II above (in the measuring cylinder) contains compound B, that is, benzaldehyde. You will be preparing dibenzalacetone from benzaldehyde. The reaction is presented below:



The density of benzaldehyde = 1.04 g cm-3 and that of acetone = 0.787 g cm-3.

Using the reaction given above and the density data, calculate the volume of acetone needed to be added for the preparation.

Volume of acetone needed for derivative preparation:

Part III: Preparation of Dibenzalacetone from benzaldehyde

To the organic layer in measuring cylinder, add the volume of acetone calculated by you with the help of a syringe supplied to you (Keep the tip of the needle of the syringe immersed in the organic layer while adding the acetone). Add 3 mL of ethyl alcohol to the same flask. In another conical flask, take 30 mL of 10 % NaOH and add 24 mL of ethanol to it. Stir the contents and keep the flask in ice bath for 5 minutes. With help of a syringe (used for transferring organic layer in part II), add half the quantity of benzaldehyde-acetone-ethanol mixture. Keep shaking the flask intermittently for about 15 minutes, without removing it from the ice bath. After this, transfer the remaining portion of benzaldehyde-acetone-ethanol mixture. Keep shaking the flask intermittently for another 15 minutes. Filter the product and wash it with 50 mL of water.

The laboratory expert will collect your products in Part II and III for drying.

Laboratory Task 3 Answersheet

Part II

2.1 Mass of the empty butter paper [g]

Mass of butter paper + compound **A** [g]

Mass of compound **A** [g]

(4 marks)

Part III

3.1 Mass of the empty butter paper [g]

Mass of butter paper + dibenzalacetone [g]

Mass of dibenzalacetone [g]

(4 marks)

3.2 (a) Colour of dibenzalacetone [ ]

(b) Appearance

Crystalline [ ] Amorphous [ ]

(Mark X in the correct box) **(1 mark)**

3.4 (a) Theoretical yield on the basis of the mass of benzaldehyde [ ]

(1 mark)

(b) The yield obtained as a percentage of theoretical yield.

[%]

3.5 Why is the reaction in **Part III** carried out in an alkaline medium?

[ ]

(1 mark)

Part IV: TLC

Procedure for TLC

Dissolve a drop of benzaldehyde in a small quantity of acetone in a sodium fusion tube. Similarly prepare a solution of your product. Obtain a TLC plate from a laboratory expert. Draw a faint line, at a distance of about 1cm from the edge of the plate. Using a thin capillary tube, place a drop of the benzaldehyde solution on the line drawn on the plate. Allow it to dry. Then in a similar manner, spot the product solution on the same plate. Take care that the two spots do not merge into one another. Allow this spot also to dry. Then place the plate in the beaker, containing the eluant (supplied to you). Cover the beaker with a watch glass, and allow the solvent to rise appreciably (approximately 1 cm away from the top). Remove the plate from the beaker and mark the solvent front **immediately**. Mark the spots after exposing the plate to UV light (laboratory expert will help you for UV chamber). Calculate the R_f values using the formula given below and record the results.

$$R_f = \frac{\text{distance travelled by the compound}}{\text{distance travelled by the solvent front}}$$

a) R_f of benzaldehyde:

(3 marks)

b) R_f of dibenzalacetone:

(3 marks)

Submit your TLC plates to the expert before leaving the laboratory.

Laboratory Task 1: Organic Synthesis

At your work bench

Part I : Preparation of derivative

▪ Beaker	1
▪ Cork	1
▪ Filter paper (circles)	4
▪ Funnels	2
▪ Glass rod	1
▪ Measuring cylinder (10 mL)	1
(25 mL)	1
▪ Syringe (1 mL)	1
▪ Spatula	1
▪ Wash Bottle	1
▪ Chemicals	5% NaOH, 25 mL
	Ethanol, 25 mL
▪ Water bath (Metal)	1
(Plastic)	1
▪ Vials	Aniline, 1 mL /vial (density – 1.021 g/L)
	Benzoyl chloride, 1.5 mL /vial

Part II: Hydrolysis of benzamide

▪ Burette	1
▪ Conical flask (100 mL)	3
▪ Funnel	2
▪ Pipette (25 mL)	1
(10 mL)	1
▪ Standard Flask (100 mL)	1
▪ Chemicals	Benzamide, 1.8 g /flask
	HCl, 50 mL
	NaOH (For blank titration)
	NaOH (For sample)
	Phenophthalein

(molarity of HCl will be supplied to you)

Laboratory Task 1 (40 Marks)

This experiment has two parts. In Part I, you will prepare benzoyl derivative of aniline. In part II you will estimate the purity of the benzamide sample supplied to you. You have three hours to complete both parts.

For the preparation of N-acyl derivatives of amines, a reaction called the Schotter-Baumann is commonly used. In this reaction, the amine in alkaline medium is reacted with an acyl chloride at low temperature. Besides many applications, such N-acyl derivatives of amines are used to characterize amines and also serve as amino group protected substrates for further selective reactions. N-benzoylaniline, known as benzanilide can be prepared by the Schotter-Bauman reaction.

Part I: Preparation of benzanilide from aniline

1 mL of aniline is supplied to you in a vial. Transfer the contents of the vial to a 100 mL conical flask. Add 20 mL of 5% NaOH solution with the help of a measuring cylinder. Using the syringe supplied, transfer the total quantity of benzoyl chloride to the conical flask. Insert the tip of the needle into the solution and slowly release the benzoyl chloride with constant shaking (**Be careful regarding the addition!)**. After the entire addition is done, cork the flask and keep it in a water bath. Shake the contents of the flask vigorously for 10 to 15 minutes. **During this period, loosen the cork twice or thrice to release the fumes.** Filter the product and wash it with 15 mL of cold water. Keep the filter paper containing the product on a tissue paper supplied to you so that the excess of water is absorbed by tissue paper.

Crystallization of benzanilide

The product is recrystallised using warm ethanol. With help of a spatula, transfer the crude product to a 100 mL beaker. Add 20 mL of ethanol to the product and heat the beaker on a water bath for 1 or 2 minutes to dissolve it. (In case the product does not dissolve, add 1 or 2 mL of ethanol). Remove the flask from water bath and allow the content to cool for 10 minutes. Filter the purified derivative. Allow the product to air dry for 15 to 20 minutes. Then transfer the content to the pre-weighed butter paper. Ask the expert to weigh your product.

Part II: Benzamide estimation

Carboxamides can be estimated by alkaline hydrolysis. A known mass of the amide is hydrolysed with an **excess** of alkali and **unconsumed** alkali is titrated against standard acid.

A consignment of benzamide was received and it was noticed that the percentage purity (percentage of desired compound in the sample) of the benzamide is not 100% as desired. The purity can be estimated using the above principal.

Blank Titration

You are given NaOH solution in plastic bottle (**Labeled for Blank Titration**). Pipette out 10 mL of this NaOH solution and titrate it with HCl solution using phenolphthalein as indicator. You are expected to take maximum three readings. In case you get two consistent reading, you need not take the third reading. This is your reading **A**.

Sample Titration

You are supplied 1.8 g of benzamide sample in a conical flask. With the help of pipette, add 25 mL of NaOH (**Labeled for Sample**) to the conical flask. Place a funnel on top of the flask and **boil** the content for 20 minutes. Remove the flask from hot plate and cool the content for 10 minutes. Transfer the content to 100 mL standard flask and dilute the solution to 100 mL using distilled water. This dilution is done so that the normality of NaOH matches with the normality of the NaOH solution used in the blank titration.

Titrate 10 mL of diluted solution against HCl using phenolphthalein indicator. You are expected to take maximum three readings. In case you get two consistent reading, you need not take the third reading. This is your reading **B**

Laboratory Task 1 Answersheet

Part I

1.1 Mass of the butter paper [g]

Mass of (butter paper + product) [g]

Mass of product [g]

(10 marks)

1.2 Colour and nature of the purified product.

[ ]

(5 marks)

1.3 Write the balanced chemical equation of reaction involved in the preparation of benzanilide, taking into account all the reagents used for synthesis.

[ ]

(1mark)

1.4 (a) Theoretical yield of the product on the basis of the mass of aniline [ ]

(1 mark)

(b) The yield obtained as a percentage of theoretical yield.

[%]

(1 mark)

1.5 At higher temperatures

i) substantial amount of benzoyl chloride will undergo hydrolysis ☐

ii) rate of reaction will increase ☐

iii) initially formed benzanilide will undergo hydrolysis ☐

iv) N,N-dibenzoylaniline will be formed ☐

(1 mark)

1.6 Can you use ammonia as a base in this reaction?

Yes ☐ No ☐

(1 mark)

Part II

Concentration of HCl: ____________________ M

Titration Readings	**Blank Titrations A**			**Sample Titrations B**		
Initial (mL)						
Final (mL)						
Difference (mL)						

Constant Reading: __________ mL Constant Reading: __________ mL

(15 marks)

1.8 Write a balanced chemical equation for the reaction involved in the titration.

(1 mark)

1.9 Calculate the amount of benzamide in mmols and in grams in the supplied sample. Hence calculate the purity of the sample supplied.

(3 marks)

1.10 With respect to ease of hydrolysis the correct statement is

i) $RCONH_2 > RSO_2NH_2 > R\text{-}COOCH_3$ ☐

ii) $RSO_2NH_2 > RCONH_2 > R\text{-}COOCH_3$ ☐

iii) $R\text{-}COOCH_3 > RCONH_2 > RSO_2NH_2$ ☐

(1 mark)

Laboratory Task 2: Iodometric Estimation

At your Work Bench

▪ Burette 50 mL	1
25 mL	1
▪ Conical flasks	7
▪ Dropper	1
▪ Funnel	2
▪ Measuring cylinder (10 mL)	1
▪ Pipette 25 mL	1
▪ Wash Bottle	1
▪ Chemicals	H_2SO_4, 3 M, 50 mL
	$KBrO_3$, 250 mL
	$Na_2S_2O_3$, 200 mL
	Sample Solution **A**
	Sample Solution **B**
	KBr, 0.5 g / vial (9 vials)
	KI, 2.5 g / vial (9 vials)
	Paraffin film strips (9 Nos)
	Starch

(molarity of $Na_2S_2O_3$ will be supplied to you)

Laboratory Task 2 (40 Marks)

Identification of phenols by iodometric method

A research student was preparing solutions of 3-methyl phenol (*m*-cresol) and 4-methyl phenol (*p*-cresol) for her project work. The student did not label the solutions and thus was confused. However, the student was clever and she adopted a particular method to identify the solutions prepared by her. She was aware that both cresols undergo brominating reaction with bromine. The required bromine can be generated using a brominating solution consisting of potassium bromate and potassium bromide. This solution on acidification liberates bromine. The bromine in turn can be estimated by its reaction with KI. The liberated iodine can be titrated with sodium thiosulphate using starch as indicator

Bottles A and B on your table contain 100 mL of sample solution **A** and sample solution **B** respectively. One solution is that of 3-methyl phenol (*m*-cresol) and another is 4-methyl phenol (*p*-cresol). You will be identifying the samples using iodometric titration based on above principle.

In this experiment you will be performing following titrations
for sample A - maximum 3 readings **for sample B - maximum 3 readings**
Blank titration - maximum 3 readings
However, if you get two consistent readings, do not take the third reading

2.1 Sample Solution A

Preparation of flasks for titrations

1. Fill the burette (25 mL) with supplied potassium bromate solution.
2. In a conical flask, pipette out 25 mL of the sample solution **A.** Then add 25 mL of the potassium bromate solution with the help of burette.
3. Add one vial of solid KBr and 5 mL of the 3 M sulphuric acid to the flask. Seal the flask using the parafilm supplied to you.
4. Prepare 2 more flasks in similar manner. Shake each flask at intervals. Keep all the three flasks aside for 20 minutes.

2.2 Titration for solution A

1. Meanwhile fill another burette (50 mL capacity) with sodium thiosulphate.
2. At the end of 20 minutes, add one vial of solid KI to the first flask. Shake the content thoroughly for 1 or 2 minutes and start titrating the liberated iodine with sodium thiosulphate solution.
3. When the colour of the solution is pale yellow, add 1 mL of starch indicator with the help of dropper. At this instance, the solution will be blue in colour.
4. Continue the titration till the solution becomes milky white in colour. Perform the titrations in similar manner for other two flasks.

2.3 Repeat the whole procedure given in 2.1 and 2.2 for sample solution B

2.4 Blank Titration

1. In a clean flask, take 25 mL of potassium bromate solution from the burette. Add one vial of solid KBr and 5 mL of the 3 M sulphuric acid to the flask.
2. Add one vial of solid KI to the flask. Shake the content thoroughly for 1 or 2 minutes and start titrating the liberated iodine with sodium thiosulphate solution.
3. When the colour of the solution is pale yellow, add 1 mL of starch indicator with the help of dropper. At this instance the solution will be blue in colour.
4. Continue the titration till the solution becomes colourless.

This reading gives **total** bromine present in 25 mL the bromate- bromide solution.

Laboratory Task 2 Answersheet

Concentration of standard $Na_2S_2O_3$: ____________ M

Sample Titrations

Total mass of sample in solution **A**: 0.140g

Total mass of sample in solution **B**: 0.140g

	Sample A			**Sample B**		
	Titration I	**Titration II**	**Titration III**	**Titration I**	**Titration II**	**Titration III**
Initial burette reading (mL)						
Final burette reading (mL)						
Difference (mL)						

Constant Reading:__________ mL Constant Reading:__________ mL

(20 marks)

Blank Titration

	Titration I	**Titration II**	**Titration III**
Initial burette reading (mL)			
Final burette reading (mL)			
Difference (mL)			

Constant Reading: __________ mL

(5 marks)

2.1 Under the given reaction conditions which of the following compounds would react with the bromine liberated from the acidified brominating solution? (Tick the correct options)

Phenylethene	☐	Cyclohexane	☐	Benzene	☐
4-methylaniline	☐	Nitrobenzene	☐	1,4-dimethylbenzene	☐

(2 marks)

2.2 Write the balanced chemical equation for the reaction involved in the titration with sodium thiosulphate (that is, reaction of iodine with sodium thiosulphate).

(1 mark)

2.3 Write the balanced chemical equation for reaction that liberates bromine. (that is, the reaction of BrO_3^- and Br^- in presence of H^+).

(2 marks)

2.4 Write balanced chemical equations for the reaction of bromine with *m*-cresol and with *p*-cresol.

(2 marks)

2.5 Calculate the amount of bromine in grams reacting with total sample **A** (that is, for 100mL of the sample solution). Show main steps of your calculations.

(3 marks)

2.6 Amount of bromine in grams reacting with total sample **B**

(1 mark)

2.7 Molar ratio of Br_2: Sample **A**

(1 mark)

Molar ratio of Br_2 : Sample **B**

(1 mark)

2.8 Hence,

Sample **A** is *p*-cresol ☐ *m*-cresol ☐ **(1 mark)**

Sample **B** is *p*-cresol ☐ *m*-cresol ☐ **(1 mark)**

Laboratory Task 1: Kinetics

At the work bench

Burette 25 mL	1
Beaker	2
Conical flasks 100mL	6
Corks (rubber)	2
Droppers	1
Funnel	1
Measuring cylinder (50 mL)	1
Pipette Bulb	1
Pipette 10 mL	1
Pipette 5 mL	1
Stopwatch	1
Wash Bottle	1
Water Bath (plastic)	1
Chemicals	0.1N H_2SO_4 - 100 mL 0.1N KI – 20 mL $Na_2S_2O_3$ - 120 ml 0.1 N H_2O_2 – 20 mL Starch indicator

(normality of $Na_2S_2O_3$ will be supplied to you)

Laboratory Task 1 (30 Marks)

To study the reaction between hydrogen peroxide and potassium iodide

In the current experiment, you will be studying the reaction between hydrogen peroxide and potassium iodide in acidic medium. Iodine is liberated in this reaction. After mixing both the reagents, fixed volume (say X mL) of the reaction mixture is drawn in another conical flask at fixed time interval. The reaction is quenched immediately using ice and the iodine present in the flask is estimated by titrating it with standard sodium thiosulphate.

Theory

When a chemical reaction follows second order kinetics, the rate of the reaction is either proportional to i) the product of the concentrations of the two reactants, or ii) to the square of the concentration of one reactant only.

If initial concentrations of the two reactants (say reactants **A** and **B**) are equal, the rate expression becomes $k = (\frac{1}{t}) \times \frac{x}{a(a-x)}$

where, k = Specific reaction rate, t = time in minutes

a = initial concentration of the reactant **A** = initial concentration of reactant **B**

x = amount reacted in time interval t

In current experiment, you will start with equal concentrations of both the reactants.

In the current experiment, the titre values are low for all the titrations. Thus, for each titration, add the titrant in drop-wise manner from beginning of the titration. Shake the content of flask thoroughly during the titration.

Procedure

Set I

1) Pipette out 10 mL of the supplied H_2O_2 solution in to a dry conical flask. Stopper the flask and immediately transfer it to a water bath. Keep the flask in water bath for at least 15 minutes. (**This is Flask I**)

2) In another conical flask, pipette out 10 mL of KI solution. To this solution, add 40 mL of 0.1M H_2SO_4 and 40 mL of distilled water using a measuring cylinder. Stopper the conical flask and place it in the same water bath. (**This is Flask II**)

3) Meanwhile rinse and fill the burette with supplied $Na_2S_2O_3$ solution.

4) At the end of 15 to 20 minutes, transfer the contents of the **flask II** to **flask I.** Note down this time as the zero time using a stop watch. Throughout the experiment **flask I** should be kept in water bath.

5) Keep another conical flask containing few pieces of ice and 2 drops of starch indicator solution ready for titration.

6) At the end of 5mins from the zero time, pipette out 5 mL of the reaction mixture and transfer it to the conical flask prepared in **step 5**. The solution will turn blue in colour.

7) Immediately titrate the solution against $Na_2S_2O_3$ with constant shaking till the solution becomes colourless. Record the burette reading.

8) Repeat the steps 5 to 7 by drawing 5 mL of the reaction mixture at t = 10, 15, 20, 25 and 30 minutes (from zero time). You have performed 6 titrations.

Set II

1) For **Flask I**, pipette out **5** mL of H_2O_2 solution.

2) In **Flask II,** pipette out **5** mL of KI solution. Add 40 mL of H_2SO_4 and 50 mL of distilled water using measuring cylinder. Keep both the flasks in water bath for 15 minutes.

3) Repeat Step No. 3 to 8 given in **Set I** to obtain six titration readings.

Laboratory Task 1 Answersheet

Normality of supplied $Na_2S_2O_3$ solution: ________________**N**

Set I :Volume of H_2O_2 = volume of KI = 10 mL

Set II :Volume of H_2O_2 = volume of KI = 5 mL

1.1 Calculate the normality of H_2O_2 in the reaction mixture that is prepared after mixing the contents of flask I and flask II. (show calculations for one set)

Set I

Similarly for **Set II**, normality of H_2O_2 : __________________________

(1 mark)

1.2 Represent the reaction taking place on mixing the contents of **flask I** and **flask II** by means of a balanced chemical equation.

(1 mark)

1.3 Similarly, write balanced chemical equation for the reaction taking place during the titration.

(0.5 mark)

1.4 For **Set I**, calculate the normality of liberated iodine in the total reaction mixture assuming the reaction is complete.

(1 mark)

1.5 For **set I**, calculate the milli-equivalence of iodine present in 5 mL of the reaction mixture at zero time (volume pipetted by you for the titration).

(0.5 mark)

1.6 Calculate the volume of supplied $Na_2S_2O_3$ that will be required to titrate the milliequivalence of iodine calculated in **1.5**. This volume you will substitute in the rate expression as **a**.

(0.5 mark)

1.7 Similarly, for **set II**, calculate the value of **a**.

(0.5 mark)

Set I Value of **a = ____________**

Time (mins)	**Titre readings(mL)**			**(a-x)**	**x/(a-x)**	**K**
	Initial	**Final**	**Difference**			
5						
10						
15						
20						
25						
30						

Set II Value of **a = ____________**

Time (mins)	**Titre readings(mL)**			**(a-x)**	**x/(a-x)**	**K**
	Initial	**Final**	**Difference**			
5						
10						
15						
20						
25						
30						

Mean k for Set I:

Set II:

1.7 Plot the graph of **x** against **t** for both sets on the **same** graph paper. Choose two points on the y-axis, $\mathbf{x_1}$ and $\mathbf{x_2}$ **-** one corresponding to **set I** and another to **set II** respectively. Points $\mathbf{x_1}$ and $\mathbf{x_2}$ should correspond to the **same fraction** of the initial concentration. Note the corresponding values of $\mathbf{t_1}$ and $\mathbf{t_2}$ from the x-axis. Using the following equation determine the order **n** of the reaction $n = 1 + \frac{(\log_{10} t_1 - \log_{10} t_2)}{(\log_{10} x_2 - \log_{10} x_1)}$

Calculation for n =

(5 marks)

1.8 Give an expression for the specific reaction rate for a second order reaction with unequal concentration of the reactants.

(1 mark)

1.9 For this experiment, the order of the reaction can also be determined by

a) half life method ☐

b) differential method ☐

c) both ☐ **(0.5 mark)**

1.10 In the above case, If one of the reactants is kept in large excess, the order of the reaction will be

a) 1 ☐

b) 2 ☐

c) zero ☐

d) unpredictable ☐ **(0.5 mark)**

Laboratory Task 2: Organic Synthesis

At the work bench

Part I: Preparation of derivative

▪ Beaker(100ml)	2
▪ Conical Flask (100mL)	2
▪ Condensor	1
▪ Dropper (plastic)	3
▪ Filter paper	2
▪ Glass rod	2
▪ Measuring cylinder (10mL)	1
▪ Round bottom flask(25 mL)	1
▪ Wash Bottle	1
▪ Chemicals	1.76g of Compound **A** 6 mL of 95% ethanol 2 mL Compound **B** 40 μL Conc.HCL 16 mL Acetone 0.65g of dimethylamine hydrochloride,

Part II: Qualitative Tests

• Tests tubes	6
• Cavity Plate	1
• Reagents (centrifuge tubes)	Sat. $NaHCO_3$ 10% aq. NaOH neutral $FeCl_3$ solution Aq. $KMnO_4$ ammoniacal solution of $AgNO_3$ NaOI 2, 4-dinitrophenyl hydrazine

Part III: TLC

- Fusion tubes with ethanol
- TLC plate
- Eluent system
- Acetophenone

Laboratory Task 2 (30 Marks)

In this task you have three parts. First part involves organic synthesis and second part involves qualitative tests and the third part involves TLC of the product. While performing the synthesis, you are expected to heat the reaction mixture for 45 to 50 minutes. After you keep the mixture for heating, you should perform Part II of the experiment (qualitative tests) and return the answer sheet to the instructor. Only after this the answer sheet for Part I will be supplied to you.

Part I: Organic synthesis

Procedure

1) You are provided with 1.76g of Compound **A** and 0.65g of dimethylamine in vials. Transfer the contents of these vials into a 25ml round bottom flask.
2) Add the entire amount of compound **B** to the round bottom flask.
3) With the help of a measuring cylinder add 4ml of ethanol and the entire amount of concentrated HCl given to you to the reaction mixture in the flask.
4) Fit a reflux condenser to the flask and reflux the mixture for 45 minutes on a preheated sand bath to 120^{o}C. **(start with part II of the experiment)**
5) After 45 minutes remove the reaction flask from the hot plate. Allow the reaction mixture to cool (around 60 ^{o}C) and transfer it to a 100 ml conical flask.
6) Add 16ml of acetone to the conical flask with stirring.
7) Cool the mixture in an ice bath to complete the crystallization.
8) Filter the product by suction filtration and wash it with 5ml of acetone. **(The instructor will help you for filtration)**
9) Dry the product for 1hr 30 minutes in an oven and weigh it.

The laboratory expert will collect your products in **Part I** for drying. After drying, the product will be given back to you for **Part III**.

Part II: Identification of functional groups in compounds A and B

You are given two compounds Compound **A** (molecular mass 30) and Compound **B** (molecular mass 120). Conduct the qualitative tests with the reagents provided to you and identify the functional groups present in compound **A** and **B**. Write your observations in the following tables.

Test	**Compound A**		**Compound B**	
	+	**-**	**+**	**-**
Sat. $NaHCO_3$				
10% aq. NaOH				
neutral $FeCl_3$ solution				
Aq. $KMnO_4$				
ammoniacal solution of $AgNO_3$				
NaOI				
2, 4-dinitrophenyl hydrazine				

Functional group	**Compound A**		**Compound B**	
	+	**-**	**+**	**-**
>C=C<				
-OH alcoholic				
-OH phenolic				
-CHO				
-CO-				
$-COCH_3$				
$-COOCH_3$				
-COOH				

(6 marks)

Part III: TLC

Procedure

Dissolve a drop of acetophenone in a small quantity of ethanol in a sodium fusion tube. Similarly prepare a solution of your product. Obtain a TLC plate from a laboratory expert. Draw a faint line, at a distance of about 1cm from the edge of the plate. Using a thin capillary tube, place a drop of the acetophenone solution on the line drawn on the plate. Allow it to dry. Then in a similar manner, spot the product solution on the same plate. Take care that the two spots do not merge into one another. Allow this spot also to dry. Then place the plate in the beaker, containing the eluant (supplied to you). Cover the beaker with a watch glass, and allow the solvent to rise appreciably (approximately 1 cm away from the top). Remove the plate from the beaker and mark the solvent front immediately. Mark the spots after exposing the plate to UV light (laboratory expert will help you for UV chamber). Calculate the R_f values using the formula given below and record the results.

$$R_f = \frac{\text{distance travelled by the compound}}{\text{distance travelled by the solvent front}}$$

a) R_f of acetophenone:

(1 mark)

b) R_f of β-dimethylaminopropiophenone hydrochloride:

(1 mark)

Submit your TLC plates to the expert before leaving the laboratory.

Laboratory Task 2 Answersheet

Compound A: ______________________________

Compound B: ______________________________

2.1 Mass of the empty butter paper [g]

Mass of butter paper + product [g]

Mass of product [g]

(9 marks)

2.2 (a) Colour of the crude product [ ]

(0.5 mark)

(b) Appearance:

Crystalline [ ] Amorphous [ ]

(0.5 mark)

2.3 (a) Theoretical yield [**(1 mark)**]

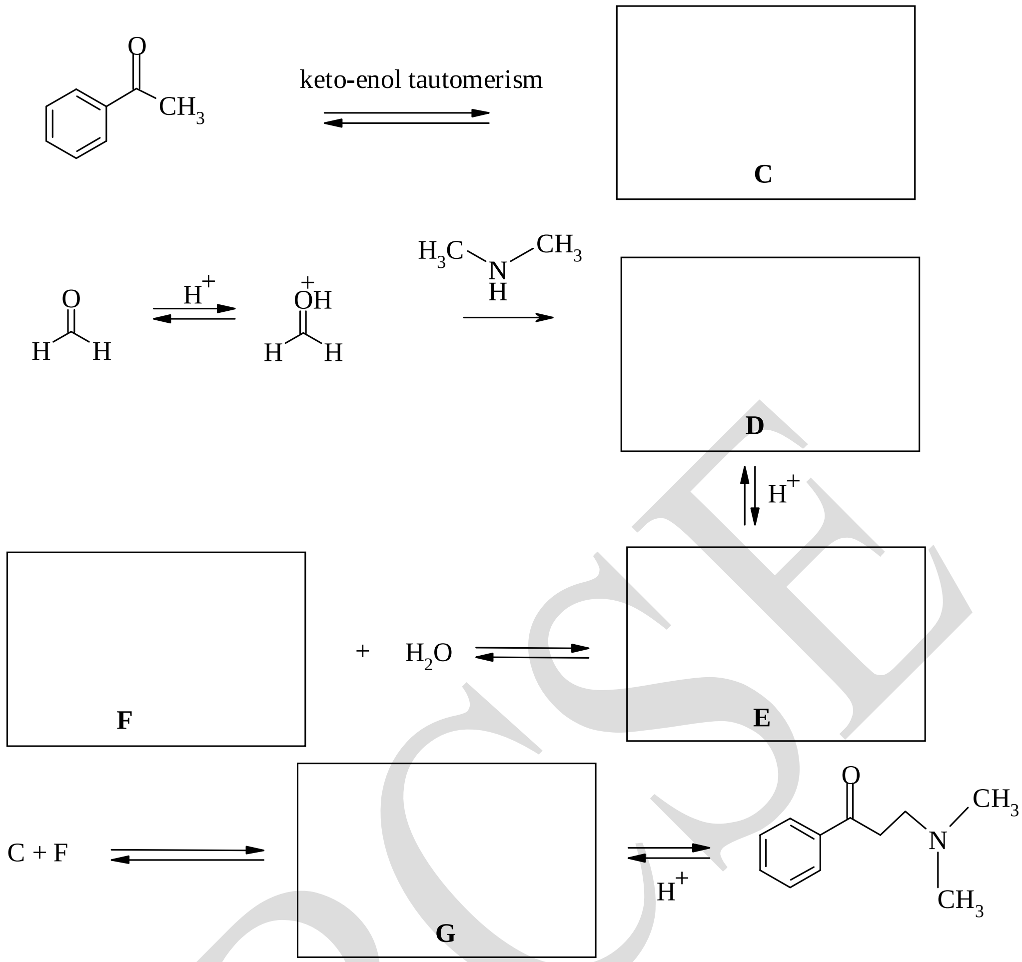
(1 mark)

(b) Calculate the yield obtained as a percentage of the theoretical yield.

[%]

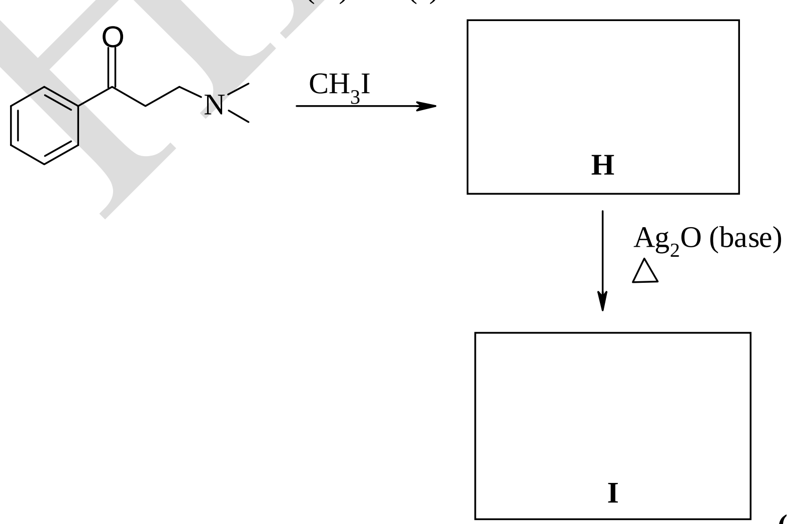
(1 mark)

2.4 The steps involved in the above synthesis are given below. Structures for some of the intermediates are not indicated in the scheme. Draw the structures of these intermediates. Also draw the curved arrows to indicate movement of electrons (wherever necessary) to depict the mechanism.



(2.5 marks)

2.6 From the Mannich base given below a very reactive molecule (**I**) can be obtained insitu. Draw the structures of (**H**) and (**I**).



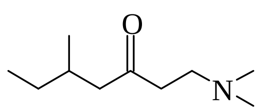
(1 mark)

2.7 Compound **I** readily reacts with cyclopentadiene. Give the structure of the product expected.

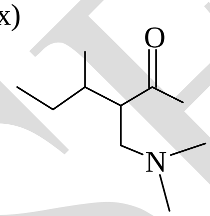
(1 mark)

2.8 Identify the molecule/s that cannot be prepared employing Mannich reaction from 4 methyl -2- hexanone. (Mark X in the correct box)

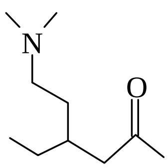
(A)



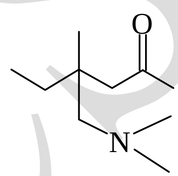
(B)



(C)

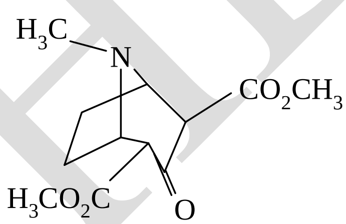


(D)



(1 mark)

2.9 Intermediate **J** used in the preparation of cocaine can be prepared by a double Mannich reaction. Identify the starting components involved in the preparation of **J** using Mannich reaction.



(2.5 marks)

2.10 The procedure adopted by you for preparation of the product using Mannich reaction is route A. Alternatively using route B the product can be obtained by the reaction of dimethylamine with **K**. Draw the structure of **K**.

(1 mark)

2.11 Route ☐ is preferred for the preparation of the product because (write alphabet A/B for the route)

(i) Route A is a 3 component reaction and not preferred over route B, which is two component reaction. ☐

(ii) Route B gives a mixture of products ☐

(iii) Route A gives a mixture of products ☐

(iv) Route B gives exclusively the product ☐

(v) Route A gives exclusively the product ☐

(Mark **X** in the correct box) **(1 mark)**

Laboratory Task 3: Estimation of Calcium

At the work bench

▪ Burette 25 mL	1
▪ Conical flasks 250mL	6
▪ Dropper	3
▪ Filter paper (circles)	4
▪ Funnel	2
▪ Glass rod	2
▪ Measuring cylinder (10mL)	1
▪ Measuring cylinder (25mL)	1
▪ Measuring cylinder (50 mL)	1
▪ Pipette 25mL	1
▪ Parafilm	3
▪ Test tubes	2
▪ Chemicals	Ca solution (supplied in 100mL flask)
	$(NH_4)_2C_2O_4$ (1.5g per vial)
	100 mL of $KMnO_4$
	20 mL of 1M H_2SO_4
	120 ml of 3M H_2SO_4
	Methyl red Indicator
	20 mL of NH_3 Solution (60% v/v)
	$(NH_4)_2C_2O_4$ Solution

(molarity of the $KMnO_4$ solution will be supplied to you)

Laboratory Task 3 (30 Marks)

Estimation of Calcium by precipitation as oxalate

You are given solution containing Ca (II). From the given solution, you will precipitate calcium as Ca-oxalate. The precipitate is then separated quantitatively from the solution by filtration and then the precipitate is re-dissolved in an acid and the oxalate content is determined by titrating it with standardized solution of $KMnO_4$.

Procedure

1. Dilute the given solution of Ca (II) to 100 mL. Pipette out 25 mL of the diluted solution in a 250 mL conical flask. To the conical flask, add 10 mL of 3 M H_2SO_4 and about 50 mL of water. Prepare another two samples in a similar manner. For each sample, perform the steps 2 to 8 given below.

   Care! Solution has 3 M H_2SO_4 that is corrosive. Be careful in handling the solution as the following steps involve hot solutions.
2. Gently heat the solution on a hot plate until the solution is just boiling.
3. Transfer the flask to your table and then **slowly** add the content of one vial of solid $(NH_4)_2C_2O_4$ with constant shaking. Keep shaking the mixture until most of the solid dissolves.
4. Add 2 to 3 drops of methyl red indicator to this solution. The colour of the solution will be pink. Now, start adding the supplied NH_3 solution in drop-wise manner until the colour changes to very light pink. If solution turns yellow, add few drops of 1 M H_2SO_4 to get the pink colour. Then once again add NH_3 to get the light pink colour in the solution (**be careful, do not inhale the NH_3 solution directly**).
5. Allow the solution to stand undisturbed for 50 to 60 minutes. Cover the flask with parafilm. Do not stir the solution during this period. At the end of this period, draw few drops of the supernatant solution in a test tube and using the supplied $(NH_4)_2C_2O_4$ solution test it for presence of Ca (II). Formation of a precipitate indicates that the precipitation of Ca (II) as oxalate is not complete. Please report to the instructor and the person will guide you.
6. If the test for Ca is negative (which indicates all the Ca (II) is quantitatively precipitated as Ca-oxalate), filter the solution. Give 2 to 3 washings to the precipitate on the filter paper, with 10 mL of water each times. At your last washing, collect few drops in a test

tube. Check this solution for the presence of oxalate using the Ca solution (present in standard flask). If needed, continue washing till there is no oxalate in the filtrate.

Care! Oxalate solutions are toxic. Do not drain these solutions in sink. Place them in a bucket near to your table.

7. Transfer the funnel containing precipitate to another conical flask. Use the glass rod to make a small hole at the bottom of the filter paper. Wash the glass rod and precipitate on the filter paper with 20 mL of water and then with 25 mL of 3 M H_2SO_4. Finally rinse the paper with another 10 to 15 mL of water.

8. Warm the solution and titrate it against $KMnO_4$ till you get a permanent light pink colour.

Laboratory Task 3 Answersheet

Molarity of $KMnO_4$ = ________________M

	Titration 1	Titration 2	Titration 3
Initial Reading (mL)			
Final Reading (mL)			
Difference (mL)			

(22 marks)

3.1 Represent the precipitation Ca (II) as calcium oxalate in the form of balanced chemical equation.

(0.5 mark)

3.2 Represent the dissolution of calcium oxalate in an acidic medium by means of a balanced chemical equation.

(0.5 mark)

3.3 Write the balanced chemical equation involved in titration.

(1 mark)

3.4 Calculate the concentration of Ca (II) in grams in the stock solution. (Show calculation for any one reading)

(4 marks)

3.5 What is the significance of the step 4 given the procedure?

(1 mark)

3.6 Why it is necessary to carry out the titration in hot condition?

(1 mark)

Laboratory Task 1: Complexometric Estimation

At the work bench

▪ Burette	(25 mL)	2
▪ Beaker	(100 mL)	2
▪ Conical flasks	(250 mL)	4
▪ Funnel		1
▪		
▪ Measuring cylinder (10 mL)		1
▪ Measuring cylinder (25 ml)		1
▪ Pipette 10 ml		1
▪ Pipette bulb		1
▪ Droppers		3
▪ pH papers		3
▪ Wash Bottle		1
Chemicals		Sample solution, 10 mL
Stoppered tubes containing		Na_2EDTA, 100 mL
		Buffer pH 10, 10 mL
		3 M NaOH, 5 mL
		Conc.HCl, 3 mL
		5 % $Na_2S_2O_3$, 35 mL

Murexide indicator is to be shared by 2 students

(molarity of Na_2EDTA will be supplied to you)

Laboratory Task 1 (25 marks)

Monel metal is an alloy of nickel and copper which is highly resistant to corrosion. It is commonly used in applications involving exposure to acids. In the current experiment, you are supplied with sample solution of Monel metal. You will be estimating the nickel and copper present in the sample by complexometric titration.

Initially in **Titration I** you will determine the content of both the ions together whereas in **Titration II** you will determine the Ni content only.

The titrant used is Disodium salt of Ethylenediamine tetraacetic acid (abbreviation: Na_2EDTA or Na_2H_2Y). Regardless of the charge on the metal ion, the titrant forms stable 1:1 complex with the metal ion.

The indicators used in the complexometric titrations are called as metal-ion indicators. They form stable complexes with the metal ions. The colour of the free indicator and that of the indictor- metal ion complex differ from each other.

Generally the pH is appropriately adjusted by using buffer solutions for the complexometric titration. In the current experiment the total ions are estimated at pH 10. By adding $Na_2S_2O_3$ and adjusting the pH only Ni can be estimated. In presence of excess $Na_2S_2O_3$, Cu (II) can not form a complex with Na_2EDTA.

Procedure

Determination of the total ion content

1. Dilute the given sample solution in your standard flask upto the mark with distilled water and shake it to homogenize it.
2. Pipette 10 mL of the diluted solution in a 250 mL conical flask and add 30 mL of distilled water.
3. With the help of the dropper, add buffer solution (pH 10) in a drop-wise manner to the flask. Keep shaking the solution during addition. Go on adding the buffer solution till your solution is cloudy and light blue in color. Continue the addition till the cloudiness just disappears.
4. Now add the murexide indicator drop wise with swirling to get a yellowish green colored solution.
5. Titrate with the given Na_2EDTA solution till you get an orange colored solution. Now add Na_2EDTA drop-wise until the color changes to violet with a single drop.
6. You are allowed to take two more readings in similar manner. Enter your readings in the answer sheet.

Determination of the Ni content

1. Pipette 10 mL of the given solution in a 250 mL conical flask and dilute it with 30 mL of water.
2. Add 3 M NaOH dropwise until the solution becomes light cloudy and light blue in color. To this now add conc. HCl drop-wise so that the cloudiness just disappears.
3. Now add 10 mL of the given 5 % $Na_2S_2O_3$ and swirl the content. Then add buffer solution (pH 10) drop-wise until the solution pH is 8 **(check with the pH paper)**. Swirl the content thoroughly.
4. Add the murexide indicator drop- wise to get an orange yellow colored solution.
5. Titrate the solution with the given Na_2EDTA until the color changes to reddish pink with no tinge of orange left in the solution. **Near the endpoint add Na_2EDTA drop wise and observe the color against a white background.**
6. You are allowed to take two more readings in similar manner. Enter your readings in the answer sheet.

Laboratory Task 1 Answer Sheet

Concentration of Na_2EDTA: ____________________ M

	Titration I			Titration II		
	Trial 1	**Trial 2**	**Trial 3**	**Trial 1**	**Trial 2**	**Trial 3**
Initial burette reading (mL)						
Final burette reading (mL)						
Volume of Na_2EDTA(mL)						

(18 marks)

1.1 Write the balanced chemical equations for the reactions of Cu (II) and Ni (II) with Na_2EDTA. (Use the symbol Na_2H_2Y for Na_2EDTA.)

(2 marks)

1.2 The reduction potential for Cu^{2+}/Cu^{1+} is 0.15 V and that for $S_4O_6^{2-}/S_2O_3^{2-}$ is 0.08 V. Explain whether Cu (II) will be reduced to Cu (I) by $Na_2S_2O_3$?

(1 mark)

1.3 For any one trial, calculate the amount of Cu (II) and Ni (II) in grams in the total diluted sample solution. (Show the main steps in your calculation).

(4 marks)

1.4 Amount of Copper (II) in grams for total diluted sample solution

Amount of Ni (II) in grams for total diluted sample solution

Laboratory Task 2 Qualitative Analysis

At the work bench

▪ Cavity plate	2
▪ Test tubes	3
▪ Droppers	9
▪ Test tube rack	1
Stoppered tubes containing	Solution 1, 5 mL
	Solution 2, 5 mL
	Solution 3, 5 mL
	Solution 4, 5 mL
	Solution 5, 5 mL
	Solution 6, 5 mL
	Solution 7, 5 mL
	Solution 8, 5 mL
	Solution 9, 5 mL
Test tubes containing	0.1 M $AgNO_3$
	1 M HCl
	3 % H_2O_2
	0.01 % phenolphthalein
	0.1 M Na_2S

Laboratory Task 2 (20 Marks)

Identification of Unknown Samples

Nine vials are kept on your table. These vials are numbered from 1 to 9. Each vial contains about 5 mL of sample solution. These unknown sample solutions are expected to be

$NaCl$	$Pb(NO_3)_2$	$Ba(OH)_2$	$Na_2S_2O_3$	
$BaCl_2$	$FeSO_4$	KI	$NaHCO_3$	NH_4SCN

Along with sample solutions, following reagents are supplied to you in test-tubes

0.1M $AgNO_3$	3% H_2O_2	0.1M Na_2S
1M HCl	0.01% phenolpthalein	

Procedure:

By performing mutual reactions and reactions with supplied reagents identify each unknown sample solution. You are expected to perform these tests in the cavity plates supplied to you. If some of the reactions are not visible, you can perform the tests in the test tubes.

If the test performed by you indicates negative result, then Mark 'X' in the appropriate cell in the table below. For positive test, write your observation in the appropriate cell

Table for tests of unknown sample solutions and reagents

Reagents/ Solution	0.1M $AgNO_3$	3% H_2O_2	0.1M Na_2S	1M HCl	0.01% Phenolpthalein
Solution 1					
Solution 2					
Solution 3					
Solution 4					
Solution 5					
Solution 6					
Solution 7					
Solution 8					
Solution 9					

Table for Mutual reactions between unknown sample solutions

Solution	**1**	**2**	**2**	**4**	**5**	**6**	**7**	**8**	**9**
Solution 1	X								
Solution 2		X							
Solution 3			X						
Solution 4				X					
Solution 5					X				
Solution 6						X			
Solution 7							X		
Solution 8								X	
Solution 9									X

Conclusion

Solution Number	Compound	Write balanced equations for positive tests. For each compound , mark the equation with (√) that confirms the presence of the sample
1		
2		
3		
4		
5		
6		
7		
8		
9		

Laboratory Task 4: Organic Synthesis

At the work bench

<u>Preparation of product</u>

▪ Beaker(100ml)	2
▪ Dropper (plastic)	2
▪ Ice bath	1
▪ Spatula	1
▪ Funnel	1
▪ Filter paper	2
▪ Glass rod	1
▪ Measuring cylinder (10mL)	2
▪ Filter papers	2
▪ Wash bottle	1
▪ Pre weighed butter paper	1
Stoppered tubes containing	$NaNO_2$ solution, 10 mL
	Conc. HCl
	10 % NaOH, 7 mL
Vials containing	Aniline, 1 mL
	β-napthol, 0.75 g
	urea

<u>For TLC</u>

▪ Beaker	1
▪ Capillary tubes	4
▪ Fusion tubes	2
▪ TLC plates	1
▪ Watch glass	1
Chemicals	Acetone

Solvent for TLC is to be shared by two students

Laboratory Task 4 30 marks

This laboratory task involves preparation of an organic dye, namely, Sudan-I from aniline. The first step of the reaction involves diazotization of aniline using sodium nitrite solution, and the second step involves coupling of the diazonium salt with β-naphthol.

Preparation of Sudan I

Procedure

a) Preparation of diazonium salt

1. A vial containing 1 mL of aniline is supplied to you. Transfer the entire content of the vial to a clean 100 mL beaker. Using a measuring cylinder, add 2.5 mL of conc. HCl and 5 mL of distilled water to this beaker. Stir the solution with a glass rod to obtain a clear solution. Cool this solution in an ice-bath for 5 to 10 minutes.
2. Chilled sodium nitrite solution is supplied to you on your table. Add 5 mL of the sodium nitrite in a dropwise manner to the above aniline solution with constant stirring. The addition should be done in cold condition only.

b) Coupling Reaction

1. In another clean beaker transfer the entire content of the vial containing β-naphthol. Add 5 mL of NaOH solution and 5 mL of distilled water. Stir well with a glass rod to obtain a clear solution. Cool this solution in an ice-bath to 0°C.
2. Add dropwise the ice cold diazotised solution (**prepared in Part a**) to the ice cold solution of β-naphthol with constant stirring.
3. Filter the precipitate using a Buchner funnel and under suction. Inform the laboratory expert when the filtration is over. After filtration, the precipitate should be handed over to the laboratory expert for drying.
4. The precipitate will be handed back to you after it is dried. Carefully transfer the product on a pre-weighed butter paper supplied to you. Take the product for weighing to the laboratory expert.
5. Record the TLC of the final product after it is weighed.

Procedure for TLC

Dissolve a pinch of Sudan I in a small quantity of acetone in a sodium fusion tube. Get a TLC plate from the laboratory expert. Draw a faint line, at a distance of about 0.5 cm from the edge of the plate. Using a thin capillary tube, place a drop of azo dye on the line drawn on the plate. Allow the spot to dry. Place the plate in a beaker, containing the solvent. Cover the beaker with a watch glass, and allow the solvent to rise appreciably (approximately 1 cm away from the top). Remove the plate from the beaker and mark the solvent front immediately. Calculate the R_f value using the formula given below and record the result in the answer sheet.

$$R_f = \frac{\text{distance travelled by the compound}}{\text{distance travelled by the solvent front}}$$

Submit your TLC plates to the expert before leaving the laboratory.

R_f for Sudan I : [ ]

(3 marks)

Laboratory Task 4

Answer sheet

4.1 The mass of the product:

(12 marks)

4.2 The calculated theoretical yield (based on aniline) in g :

(1 mark)

4.3 The yield obtained as a percentage of the theoretical yield:

(1 mark)

4.4 Colour of the product obtained:

(a) Dark Brown

(b) Yellow

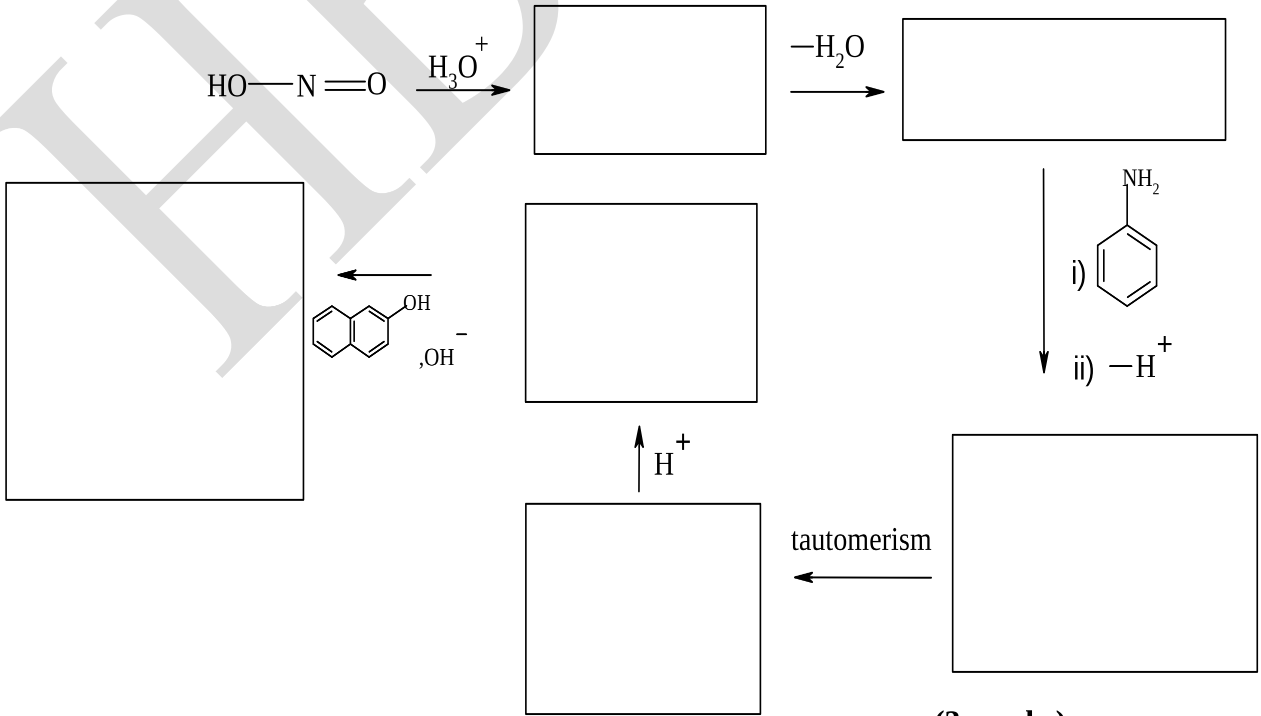
(c) Orange red

(d) Red

(e) Any other

(5 marks)

4.5 Mechanism of reactions involved in synthesis of Sudan-I is given below. Draw the structures of intermediates and Sudan-I.

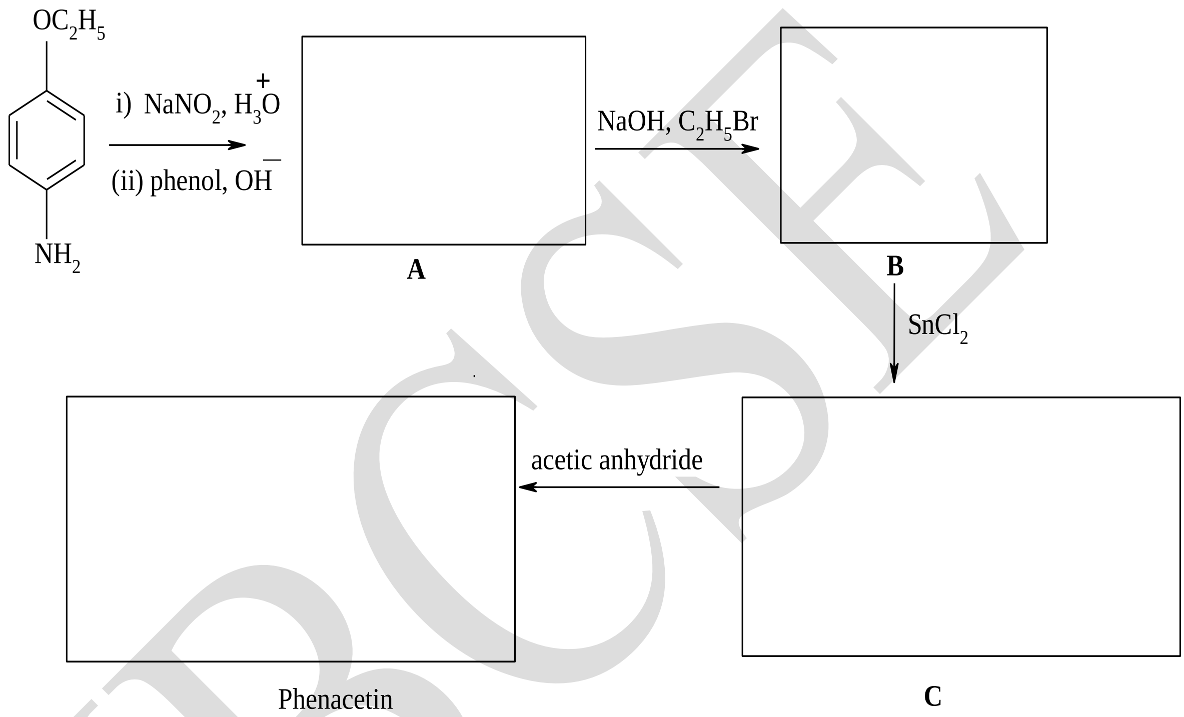


(3 marks)

4.6 Azo compounds can be reduced to amines by a variety of reagents. $SnCl_2/HCl$ is one of them.

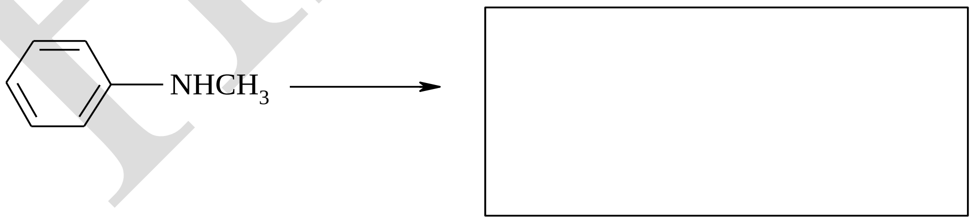
$$Ar-N=N-Ar' \xrightarrow{SnCl_2/HCl} ArNH_2 + Ar'NH_2$$

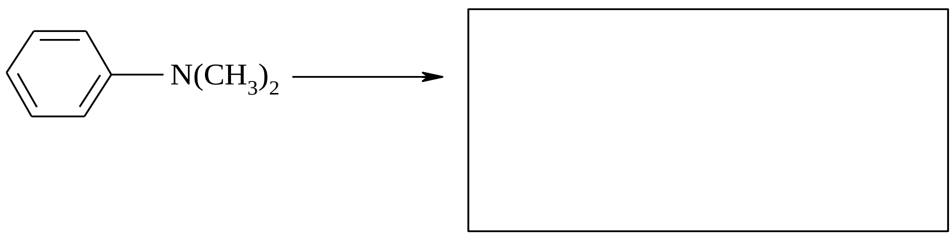
This reduction can be useful in the synthesis of Phenacetin (an analgesic). Give the structure of phenacetin and the intermediates **A**,**B** and **C**.

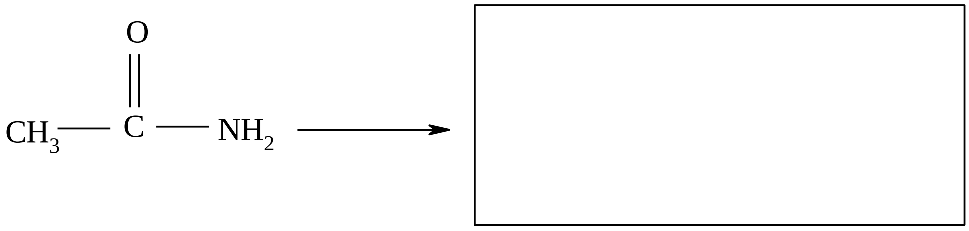


(2.5 marks)

4.7 Draw the structures of the products obtained when the following compounds are treated with $NaNO_2/HCl$ at 0^oC.

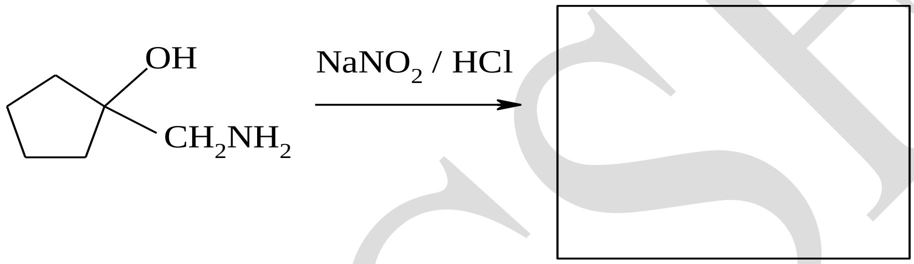
(i) 

(ii) 

(iii) 

(1.5 marks)

4.8 Treatment of the following amino compound with $NaNO_2$/HCl at 0 to 5°C gives compound **E** which gives a positive 2,4-DNP test. Draw the structure of **E**



E

(1 mark)

Laboratory Task 5: Analysis of antacid tablet

At the work bench

▪ Burette (25mL)	2
▪ Conical flasks (250mL)	4
(100 mL)	1
▪ Dropper	2
▪ Funnel	1
▪ Filter paper	1
▪ Measuring cylinder (10ml)	1
▪ Measuring cylinder (25ml)	1
▪ Pipette 10 ml	1
▪ Pipette bulb	1
▪ pH papers	3
▪ Standard flask (100 mL)	1
▪ Wash bottle	1
Stoppered tubes containing	Sample, 0.5 g
	Na_2EDTA
	Buffer pH 10, 120 mL
	Triethanolamine (TEA), 10 mL
	$ZnSO_4$ solution, 20 mL

Calmagite indicator is to be shared among two students

Hot plate is to be shared among two students

(molarity of Na_2EDTA and $ZnSO_4$ will be supplied to you)

Laboratory Task 5 (30 Marks)

Analysis of an antacid drug for its aluminum and magnesium content

Antacids are useful in relieving acid indigestion and sour stomach. They can be generally divided into two classes: i) Chemical antacid that works by chemical neutralization of gastric acid. For example, sodium bicarbonate (*eno* powder that you take), ii) adsorptive antacid act by adsorbing the acid, including aluminium and magnesium salts. The former category shows rapid action but sometimes can cause a condition of *acid rebound*, a condition where the gastric acid concentration is much more after the drug effect is stopped. The latter category generally does not cause a rebound effect.

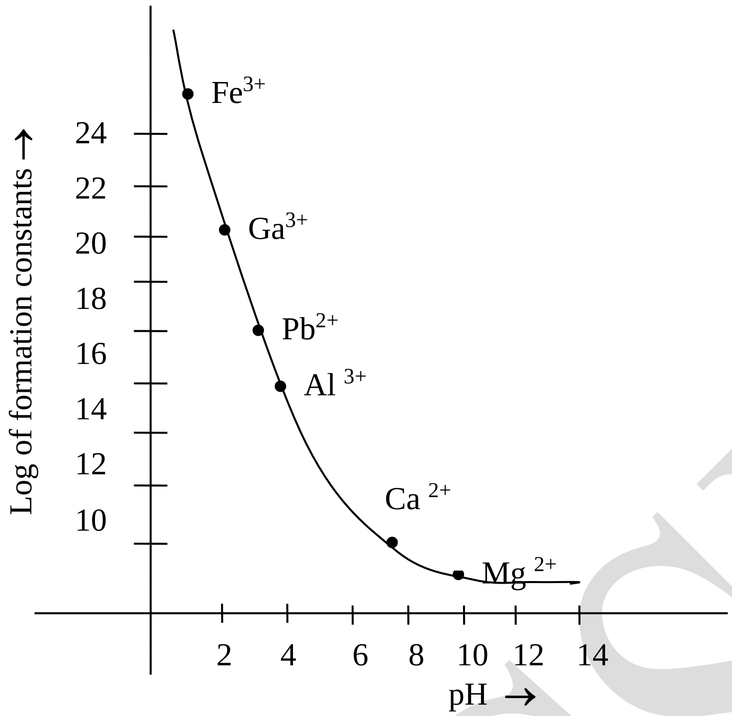
In this experiment you will estimate the amount of Al and Mg ions of a commercial antacid tablet. This drug contains Al(III) as Aluminium hydroxide and Mg (II) as Magnesium hydroxide. Initially, you will dissolve the drug with an acid and prepare a solution of known dilution. Then a fixed volume of this solution will be taken for titration purpose. You will be determining Al and Mg using complexometric titration.
Initially in **Titration I** you will determine the Al and Mg content together whereas in **Titration II** you will determine the Mg content only.

Theory

In the current experiment, the titrant used is Disodium salt of Ethylene diamine tetraacetic acid (abbreviation: Na_2EDTA or Na_2H_2Y). Regardless of the charge on the metal ion, the titrant forms stable 1:1 complex with the metal ion.

The indicators used in the complexometric titrations are called as the metal-ion indicators. They form stable complexes with the metal ions. The colour of the free indicator and that of the indictor- metal ion complex differ from each other. The metal-ion indicators themselves are either weak acids or weak bases.

The graph given below is the graph of log of the formation constants of the complexes of the metal ions with Na_2EDTA, as a function of pH. The metal ions you will be estimating are indicated on the graph.



Procedure

Preparation of sample for analysis

A vial containing the sample is supplied you. Carefully empty the entire content of the vial in a 100 mL conical flask. Add 50 mL of water and 3 mL of 6 M HCl solutions. Transfer the flask to a hot plate and gently boil the solution for 20 to 25 minutes. At the end of the stated time interval remove the flask from the hot plate and allow it to cool for 5 minutes. Filter the solution and collect the filtrate in 100 mL standard volumetric flask.

Filtration of the solution

Fold the circular filter paper given to you inside the funnel and slightly wet the paper so that it adheres to the funnel properly. Place the funnel in a 100 mL standard volumetric flask. Start filtering the solution with the help of a glass rod otherwise the solution will trickle down to the working platform. Use minimum amount of water to transfer the entire content of the conical flask to the volumetric flask. Remove the funnel and then dilute the solution upto the mark with distilled water.

Determination of the total Al and Mg content (Titration I)

1. Pipette 10 mL of the diluted solution in a 250 mL conical flask and add 30 mL of water. Then add 25 mL of buffer solution (pH 10) followed by 40 mL of supplied Na_2EDTA solution.

2. Boil the mixture for 5 minutes on a hot plate. Remove the flask from the hot plate (use gloves to hold the hot flask) and add to it 5 drops of *Calmagite* indicator. The solution should turn blue. If not, then add another 5 mL of Na_2EDTA solution and boil until colour changes to blue. Then titrate the hot solution against standardized $ZnSO_4$ solution till the colour changes to purple.

3. Take at least two more readings in similar manner. Enter your reading in the answer sheet.

Determination of the Mg content (Titration II)

1. Pipette 10 mL of the given solution in a 250 mL conical flask and dilute it with 30 mL of water.

2. Add 25 mL of pH 10 buffer followed by addition of 3 mL of Triethanolamine. Keep shaking the solution thoroughly for 1 minute and allow it to stand for a while. You will get almost a clear solution at this point.

3. Add 5 drops of *Calmagite* indicator and swirl the content. The solution should be wine red in colour.

4. Titrate the solution with the given standard Na_2EDTA solution until the colour changes to pure blue at the end point. **Perform the titration as quickly as possible.**

5. Take at least two more readings in a similar manner. Enter your results in the answer sheet.

Laboratory Task 5 **Answersheet**

Concentration of Na_2EDTA solution: ____________ M

Concentration of $ZnSO_4$ solution: ____________ M

	Titration I			Titration II		
	Trial 1	**Trial 2**	**Trial 3**	**Trial 1**	**Trial 2**	**Trial 3**
Initial burette reading (mL)						
Final burette reading (mL)						
Volume of Na_2EDTA(mL)						

(20 marks)

5.1 Write the balanced chemical equations for the reactions of Al and Mg with Na_2EDTA. (Use the symbol Na_2H_2Y for Na_2EDTA.)

(1 mark)

5.2 Calculate the Aluminum hydroxide and Magnesium hydroxide content in grams in the total diluted sample. Show your calculations for any one set of reading. (Show main steps in your calculation).

(4 marks)

5.3 The colour change at the end point (blue to purple) in the **Titration I** is due to [Mark **X** in the correct box.]

(a) the formation of the metal-indicator complex. ☐

(b) the release of the free indicator from the metal-indicator complex. ☐

(c) the formation of metal-EDTA complex. ☐

(1 mark)

5.4 If you have to determine the Aluminium content **alone** then at what optimum pH the titration should be performed? Between Magnesium and Aluminum which forms stronger complex with Na_2EDTA.

(2 marks)

5.5 The solution needs to be boiled in **Titration I** whereas Titration II is performed at room temperature. This indicates [Mark **X** in the correct box.]:

(a) the formation of Aluminium-EDTA complex is kinetically slow ☐

(b) the formation of Magnesium-EDTA complex is kinetically slow ☐

(c) the formation of indicator-EDTA complex. is kinetically slow ☐

(1 mark)

5.6 The procedure states that **Titration II** should be performed as rapidly as possible. This indicates [Mark **X** in the correct box.]:

(a) the Aluminium-TEA complex is very stable ☐

(b) the Magnesium-EDTA complex is not stable ☐

(c) the indicator-Na_2EDTA complex. is stable ☐

(1 mark)