Laboratory Task: Inorganic Preparation, Estimation and qualitative tests

Part 1: Overview of glassware and chemicals required for the experiment

Preparation of complex (Part A)

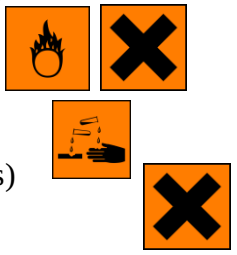
- Beakers 100 mL — 2
- Glass rod — 1
- Ice Bath — 1
- Measuring cylinder 10mL — 1
- Chemicals

$Fe(NO_3)_3.9H_2O$	3.000 g/vial (1 vial)
$K_2C_2O_4$	8.000 g/vial (1 vial)

Analysis of the complex (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)

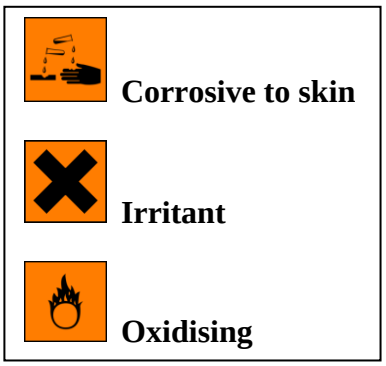
- Cavity Plate — 1
- Droppers — 4
- Chemicals

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)



Part 2: Introduction to the experiment and Procedure

Introduction

Transition elements are known to form variety of co-ordination complexes. These complexes are often highly colored and depending on the ligand, the color of a given complex varies for a given transition element.

For example Fe (II) shows a variety of colors with different ligands like, oxalate, thiocyanate, cyanide, chloride, water etc. Some of these complexes are shown below

Potassium ferrioxalate

Potassium ferricyanide

Prussian blue

In this experiment you will prepare a co-ordination complex of Fe (III) and oxalate in part A (image 1). In part B of the experiment, you will be analyzing the given complex for its iron and oxalate contents using titration technique. The molecular formula of the prepared complex is $K_aFe_bOx_c.dH2O$.

In part C, you will be performing some qualitative tests with solutions of iron (III) nitrate and the complex. 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 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 an ice bath and keep it 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: Analysis of unknown co-ordination complex of iron

Determination of the oxalate content

A sample of 0.250 g will be weighed in duplicate for analysis, in the presence of the laboratory expert.

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 - 80^0C.
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.

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 1**, iron exists as $[Fe(H_2O)_6]^{3+}$.**

Carry out the following tests and report your observations.

Solution tested	**Reagent 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.

(2 marks)

Part 3: Observations, Calculations and Questionnaire

Typical results have been mentioned here.

Concentration of $KMnO_4$: 0.0196 M

1

	Trial I		**Trial II**	
	Titration 1	**Titration 2**	**Titration 1**	**Titration 2**
Initial burette reading (mL)	00.0	00.0	0.00	00.0
Final burette reading (mL)	30.7	5.4	30.7	5.4
Volume of $KMnO_4$ (mL)	30.7	5.4	30.7	5.4

(16 marks)

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

HC_2O_4 [X] $HC_2O_4^-$ [ ] $C_2O_4^{2-}$ [ ]

(0.5 mark)

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

$2KMnO_4 + 5H_2C_2O_4 + 3H_2SO_4 \rightarrow K_2SO_4 + 2MnSO_4 + 8H_2O + 10CO_2$

or equivalent ionic equation

(0.5 mark)

4 Write the reactions that take place

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

b) when the solution is boiled.

a) $Zn + 2H^+ \rightarrow Zn^{2+} + H_2$ or

b) $Zn + 2Fe^{3+} \rightarrow Zn^{2+} + 2Fe^{2+}$

(1mark)

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

$MnO_4^{1-} + 8H^+ + 5Fe^{2+} \rightarrow Mn^{2+} + 5Fe^{3+} + 4H_2O$
$MnO_4^{1-} + 8H^+ + 5e^- \rightarrow Mn^{2+} + 4H_2O$
$5Fe^{2+} \rightarrow 5Fe^{3+} + 5e^-$
$MnO_4^{1-} + 8H^+ + 5Fe^{2+} \rightarrow Mn^{2+} + 5Fe^{3+} + 4H_2O$

(1 mark)

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

(2 marks)

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

(3 marks)

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

(2 marks)

10. a) Mass of the pre-weighed butter paper

Mass of product + butter paper	
Mass of product	

b) Colour of the product

11. Write the balanced chemical equation/s for the reaction/s involved in formation of the complex.