

CHEMISTRY LABORATORY RECORD

Name

Roll No.....Batch.....Reg. No.....

*Certified that this is the bonafide record of work done by
.....during the monsoon semester 2012-2013.*

Date:

Faculty-in-charge

**Department of Chemistry
National Institute of Technology Calicut
Kerala-673601**

General Instructions

1. Before you come to the laboratory, prepare for the day's work. Understand the theory behind the experiment which you propose to carry out. You will turn out to be a better analyst if you have a full understanding of what you are doing. Carrying out instructions mechanically does not produce a good scientist/engineer.
2. You should understand and appreciate the experiments to be performed. If in doubt or difficulty seek help.
3. When you come to the laboratory, provide yourself with a box of matches and two pieces of cloth a rough cloth to wipe off liquid split upon the work-bench, and another for drying the inside of the test tubes and vessels used in qualitative analysis. Always wipe off quickly liquids that are split on the work-bench or on the filter-stand when filtering, paying special attention to strong acids which should be neutralized with sodium carbonate solution before removal.
4. Wear the institute over coat which is available in the co-operative store or extension counter.
5. Keep the work-bench and sink neat and clean. Don't allow filter paper, broken bits of glass, sticks of matches, charcoal etc. to lie on the table or sink. Put these things in the bowls placed on the table.
6. Keep the apparatus clean and properly arranged on the work-bench.
7. Strong acids used for conducting experiments should be diluted before being poured into the sink.
8. If any piece of apparatus is broken report at once to the Technical staff who is in charge of the class.
9. Once you have taken a reagent from a bottle, never pour it back even if there is some excess.

General Instructions

10. Replace reagent bottles in their proper places at once after use and see that they are stoppered. Never remove more than one reagent bottle at a time.
11. Never handle chemicals with your finger; always use a spatula.
12. Use only the minimum possible quantity of chemicals for reactions.
13. When the burner is not being used, close the gas tap.
14. Close the water tap immediately after use; do not waste water.
15. There is always risk of fire in a chemical laboratory. A student should be on his/her guard to avoid every kind of fire hazard.
16. After the class, before you leave the laboratory, wash every apparatus clean, wipe the table and keep everything in the 'as before' condition.

Records

Every student must have **only one record note book**. This record book is intended for entering his/her observations while the experiment is being done. This must be done carefully and neatly as one goes along with the work and should never be postponed to the last minute. And the sufficient care must be given to maintain this record neat and clear.

In quantitative experiments, write the experimental portion on the right hand page and enter the data and calculation on the left hand page. The record of every exercise (i.e., day's work) must be serially numbered and dated. As soon as the allotted work is over, submit the record note book to the Faculty member in charge of the class and get it signed. The evaluation is done based on the percentage of error in each experiment. The experiment must be carried out with appropriate care and attention. The final marks will be assigned based on the errors in each experiment and test which is conducted after the two cycles of the experiments.

Take Care of Accidents

In case of accidents, while inside the laboratory, call or notify the member of the staff who is in charge of the day's work.

1. Clothing on fire: Wrap the person in a heavy blanket or wet gunny bag.
2. Fire while burning of reagents: Extinguish gas burners in the vicinity. Small fires in flasks or beakers may be put out covering the opening with asbestos sheets, wire gauze or wet towel.
3. Acid on clothing: Neutralize with dilute ammonium hydroxide. Then wash thoroughly with water
4. Alkali on clothing: Neutralize with dilute acetic acid. Then wash thoroughly with water.
5. Acid in eye: Wash thoroughly and profusely with running water. Bathe the eye with a 2% sodium bicarbonate solution, using an eye cup. Dry with sterile gauze and put several drops of olive oil into the eye.
6. Alkali in eye: Wash thoroughly with running water and the eye-lids should be kept widely opened especially when caustic alkalies have entered the eye. Bathe with boric acid solution, dry and add a drop of olive oil into the eye.
7. Ordinary heat burns: Don't use water. Apply sodium bicarbonate vaseline paste or burnol and consult our resident doctor.
8. Acid burns: Wash first with running water and then with sodium bicarbonate solution. Cover with solid sodium bicarbonate for 10 minutes. Wash off and apply carron oil (a mixture of equal parts of lime water and linseed oil).
9. Alkali burns: Wash first with running water and then with boric acid solution. Cover with powdered boric acid for 10 minutes. Wash off and apply sodium bicarbonate vaseline paste.
10. Bromine burns: Wash with petrol and apply olive oil.
11. Bromine or chlorine poisoning: Inhale ammonia vapour; remove to open air.
12. Cuts: Remove particles or glass, if any are present. Wash the wound with water. Then apply tincture of iodine and cover with a sterile bandage.

For all except minor injuries our Resident Doctor should be consulted as soon as possible.

Poisoning

- a) *By strong acid.* Give plenty of water or milk. Then give two table spoons of lime water.
- b) *By caustic alkalies.* Give plenty of water or milk. Then give orange or lemon juice.
- c) *By copper sulphate.* Immediately give a table spoon of common salt or zinc sulphate in a tumbler of warm water.
- d) *By mercury salt.* Give plenty of warm water or warm milk and make vomiting.

Inhalation of Gas

- a) *By chlorine or bromine.* Inhale ammonia or alcohol vapour.
- b) *By carbon monoxide.* Inhale open fresh air.
- c) *By arsine or phosphine.* Inhale open fresh air.
- d) *By nitrous fumes.* Inhale plenty of fresh air and then steam.
- e) *By chloroform.* Inhale plenty of fresh air. Administrate hot and cold douches.
- f) *By hydrogen sulphide or hydrogen cyanide.* Inhale ammonia, give artificial respiration; administer warm and cold douches to head.

List of Experiments

- 1) Potentiometric estimation of chloride ion in drinking water
- 2) Molecular weight determination of Polymer
- 3) Conductometric titration of an acid / mixture of acids
- 4) Determination of strength of an acid using a pH meter
- 5) Determination of specific rotation and strength of unknown sugar solution polarimetrically
- 6) Estimation of magnesium
- 7) Colorimetric estimation of copper in brass
- 8) Determination of Iron in Haematite Ore
- 9) Phase rule: Two component systems
- 10) Analysis of organic compounds

Observations:

S. No.	Vol. of Cl^- added	ΔV	Emf (mV)	ΔE	$\Delta E/\Delta V$

Calculations:

Concentration of silver nitrate solution (N_1) =

Volume of silver nitrate solution (V_1) = 50 mL

Volume of chloride ion solution used = mL

$$N_1 V_1 = N_2 V_2$$

Conc. of the solution (N_2) =

Strength of NaCl = $N_2 \times \text{Equivalent weight of NaCl}$

$$= N_2 \times 58.5$$

Amount of Cl^- in the given solution = $N_2 \times 35.5$

$$= \dots \times 35.5$$

$$= \dots \text{ g/litre}$$

Experiment No.....

Date

Potentiometric estimation of chloride ion in drinking water

Objective:

To estimate chloride ions in the given water sample, potentiometrically.

Principle:

When AgNO_3 solution is treated with NaCl solution from the burette the concentration of Ag^+ ions in the solution goes on decreasing as Ag^+ is removed as AgCl . Thus silver-electrode is reversible with respect to chloride ions (indicator electrode). Therefore as chloride ion concentration changes, the potential of the electrode changes. It is coupled with a reference electrode, and the emf of the galvanic cell so formed is measured. From the volume of silver nitrate solution used and the change in emf, the quantity of chloride ions present in the given solution can be determined.

Procedure:

Pipette out 50 mL of AgNO_3 solution into a clean 100 mL beaker and dip the electrodes properly into it. Add 0.2 mL of the chloride ion solution from a burette. Stir well and note the emf of the cell. Continue adding 0.2 mL each of Cl^- ion solution. Plot a graph with $\Delta E/\Delta V$ along Y-axis and volume of chloride solution added along X-axis

Result:

The amount of chloride ions in the given solution = g L^{-1}

Experiment No.....

Date

Molecular Weight Determination of Polymer

Objective:

To determine the molecular weight of a polymer, e.g., polyvinyl alcohol, polystyrene etc, from viscosity measurements.

Theory:

The viscosity of a dilute (conc. less than 1%) polymer solution is related to the molecular weight of the polymer by the Mark-Houwink equation, $[\eta] = K\bar{M}^a$ is the intrinsic viscosity and \bar{M} is the molecular weight of the polymer, 'K' and 'a' are constants for a given solvent-polymer system at a particular temperature.

Intrinsic viscosity is defined by

$$[\eta] = \lim_{c \rightarrow 0} \frac{\eta_{sp}}{c}$$
$$= \lim_{c \rightarrow 0} \frac{\eta_{soln} - \eta_{solvent}}{\eta_{solvent} \times c}$$

Where η_{sp} the specific viscosity of the polymer solution and c is the concentration in g of polymer in 100 mL of the solvent. The specific viscosity η_{sp} is related to the relative viscosity η_r by the equation $\eta_{sp} = \eta_r - 1$; i.e., $\eta_{sp} = (\eta/\eta_0) - 1$, where η is the viscosity of the solution and η_0 is the viscosity of the solvent. The intrinsic viscosity is obtained by plotting η_{sp}/c i.e., reduced viscosity or $\frac{\ln \eta_r}{c}$ i.e., inherent viscosity against concentration, c (g/100ml) and extrapolating to zero concentration.

Apparatus:

Ostwald viscometer, Stopwatch.

Procedure:

Prepare five sets of solutions containing 0.8g, 0.6g, 0.4g and 0.2g of the polymer in 100 mL each of pure solvent (water) in 5 standard flasks. Introduce about 20 ml of solution in to a dry viscometer and find out the flow time. Repeat the same for other set of solutions and also for pure solvent. From the flow time of the solution and that of the pure solvent at that temperature we can calculate the relative viscosity, η_r of each solution and hence the η_{sp} . Now plot η_{sp}/c against c and $\frac{\ln \eta_r}{c}$ against c and extrapolate the curve to zero concentration. The intercept will give the value of intrinsic viscosity. Now knowing the value of K and 'a' for that particular solvent- polymer system, the value of the intrinsic viscosity $[\eta]$ can be obtained. We can now calculate the molecular weight of the polymer by the equation $[\eta] = K \bar{M}^a$

Preparation of polyvinyl alcohol solution (Typical example)

Prepare a stock solution of polyvinyl alcohol (PVA) by dissolving 2g of PVA in 50 mL of boiling water. It is then cooled, transferred quantitatively to a 100 mL standard flask and made up to 100 mL to obtain a 2 % PVA solution. From this stock solution, prepare 0.8, 0.6, 0.4 and 0.2 % PVA solutions by pipetting appropriate amounts of stock PVA solution and diluting.

Result:

The molecular weight of the given polymer = gmol^{-1}

Observations:

S. No.	Volume of alkali (mL)	Conductivity (mho)	S. No.	Volume of alkali (mL)	Conductivity (mho)
1			26		
2			27		
3			28		
4			29		
5			30		
6			31		
7			32		
8			33		
9			34		
10			35		
11			36		
12			37		
13			38		
14			39		
15			40		
16			41		
17			42		
18			43		
19			44		
20			45		
21			46		
22			47		
23			48		
24			49		
25			50		

Experiment No.....

Date

Conductometric titration of an acid / mixture of acids

Objective:

To determine the strength of the given acidic solution using standard alkali solution, conductometrically.

Theory:

The variation of conductivity by the addition of a titrant is measured. The plot consists of two curves which intersect at a particular point known as end point or equivalent point. The method can be employed even to very dilute solutions.

Consider the acid is taken in a beaker and the base in the burette. The solution contains H^+ and A^- at first. The initial conductivity of the solution is mainly due to H^+ ions. As alkali is added, the H^+ ions are removed as unionized water.



Therefore, the conductivity decreases. At the neutralization point, the solution has a considerably lower conductivity than the original value. If a drop of alkali is added after neutralization point, the conductivity increases due to the high mobility of OH^- . As more and more alkali is added, the conductivity goes on increasing continuously. Hence on plotting conductivity values against the volume of the titrant added, two straight lines are obtained and the point of intersection gives the equivalence point.

Procedure:

Pipette out 50 mL of given acid solution in a clean dry 100 mL beaker. Submerge the conductivity cell, after proper washing with distilled water. The given alkali solution is

Calculations:

Volume of alkali $V_1 = \dots\dots\dots$ mL

Normality of alkali $N_1 = \dots\dots\dots$

Volume of acid $V_2 = \dots\dots\dots$ mL

Normality of acid $N_2 = \frac{V_1 N_1}{V_2} = \frac{\dots\dots\dots \times \dots\dots\dots}{\dots\dots\dots} = \dots\dots\dots$

Strength of given acid $= \text{Normality of acid } (N_2) \times \text{Mol. Wt}$

$= \dots\dots\dots \text{ gL}^{-1}$

taken in the burette. Titrate it against acid solution. Note the conductance after every addition of 0.2 mL. increments. Plot a graph between conductance versus volume of the alkali added and find out the strength using normality equation.

Result:

The strength of the given acid solution = gL^{-1}

Observations:

S. No.	Volume	pH	$\Delta\text{pH}/\Delta V$

Calculations:

Normality of alkali =

Volume of alkali pipetted (V_1) = 50 mL

Volume of acid corresponding to the equivalence point from the graph (V_2) = mL

$$\begin{array}{rcl} \text{N}_1\text{V}_1 & = & \text{N}_2\text{V}_2 \\ \text{(alkali)} & & \text{(acid)} \\ \text{N}_1 \times 50 & = & \text{N}_2 \times \text{V}_2 = \text{N}_2 \times \dots\dots\dots \end{array}$$

$$\therefore N_2 = \frac{N_1 \times 50}{V_2} = \dots\dots\dots$$

Strength of the acid solution = Mol. Wt of acid $\times N_2$ =

Result:

The strength of the given acid solution = $\quad \text{gL}^{-1}$

Experiment No.....

Date

Determination of strength of an acid using a pH meter

Objective:

To determine the strength of the given acid by titrating it against alkali using a pH meter.

Theory:

When an alkali is added to an acidic solution the pH of the solution increases slowly, but at the vicinity of the equivalence point, the rate of change of pH of the solution is very rapid. From the sharp break in the curve, we can find the equivalence point, from which the strength can be calculated.

Procedure:

Estimation of the given acid:

Pipette 50 mL of the standard alkali into the beaker. The electrodes are properly dipped in the solution. Note the pH of the solution. Now add 0.2 mL of acid from the burette to the beaker. Stir the contents well. Again note the pH of the solution. Continue addition of 0.2 mL of the acid into it and each time the respective pH is also noted. Graphs are plotted with pH vs. volume of acid and $\Delta\text{pH}/\Delta V$ vs. volume of acid (X axis) added. From the curves the end point is noted.

Result:

The strength of the given acid solution = gL^{-1}

Observations:

S. No.	Concentration in g/100 mL	Angle of rotation Observed (θ)	$[\alpha]_D^T = 100 \theta / \ell c$
1			
2			
3			
4			
5			
Unknown			
Average value $[\alpha]_D^T =$			

Results:

Specific rotation $[\alpha]_D^T =$

Unknown concentration from the graph =

Experiment No.....

Date

Determination of specific rotation and strength of unknown sugar solution polarimetrically

Aim:

To determine the strength and specific rotation of unknown sugar solution.

Principle:

The sucrose solution rotates the plane polarized light to the right. The optical rotation depends on the concentration of the sucrose solution by the equation.

$$[\alpha]_D^T = 100 \theta / \ell c$$

Where $[\alpha]_D^T$ is specific rotation at temperature, T and source light Na - D line.

‘ θ ’ is the observed optical rotation in degrees.

‘ ℓ ’ is cell path length in decimeters.

‘c’ is concentration in g dL⁻¹

Procedure:

Prepare different solutions of concentrations (40 %, 30 %, 20 %, 10 %) by using given 50 % sugar solution. Fill the polarimeter tube with distilled water and the zero reading of polarimeter is determined. Water is removed; the tube is dried and filled with 50% sugar solution and find the angle of rotation. Repeat the same procedure for all the other solutions including unknown solution. A graph is plotted with angle of rotation (θ) on Y axis and concentration on X axis. From the graph concentration of unknown solution is calculated.

Result:

1. Concentration of given sugar solution =
2. Specific rotation of sugar solution $[\alpha]_D^T$ =

Observations:**(i) Standardisation of EDTA:**

S. No.	Vol. of ZnSO ₄ Solution (V ₁)	Burette reading		Vol. of EDTA used (V ₂)
		Initial	Final	

(ii) Estimation of Magnesium:

S. No.	Vol. of Mg ²⁺ Solution (V ₃)	Burette reading		Vol. of EDTA used (V ₄)
		Initial	Final	

Experiment No.....

Date

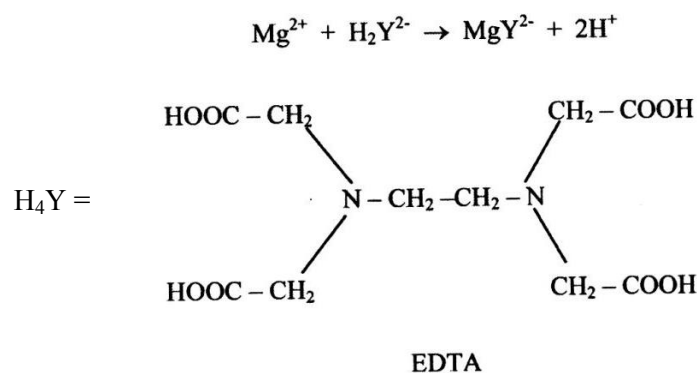
Estimation of Magnesium

Objective:

Estimate the amount of magnesium present per litre of the given solution of magnesium sulphate.

Theory:

Magnesium ions form complexes with EDTA (Ethylenediaminetetraacetic acid) which can be represented by the equation.



Eriochrome black T is used as an indicator, which gives a wine red colour in the presence of metal ions at the pH range of 7-11. At the end point all the metal ions are converted to metal-EDTA complex and the free indicator is liberated which has a blue colour. Using std. EDTA solution the given magnesium solution is estimated.

Procedure:

1. Preparation of standard ($\cong 0.025 \text{ M}$) ZnSO_4 solution

About 0.72 g of AR $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$ is accurately weighed and transferred to a 100 ml standard flask. It is dissolved in distilled water, diluted and made up to the mark.

2. Standardisation of EDTA

A burette is set with EDTA solution. 20 ml of standard ZnSO_4 solution is pipetted into a conical flask. Then 2 ml of buffer solution of pH 10 and a pinch of Eriochrome black T indicator are added to this solution in conical flask. The solution is shaken thoroughly and

Calculations:

$$\text{Mass of ZnSO}_4 \cdot 7\text{H}_2\text{O in 100 ml} = \dots\dots\dots \text{ g}$$

$$\begin{aligned}\text{Molarity of ZnSO}_4 \text{ solution (M}_1\text{)} &= \frac{\text{Mass per litre}}{\text{Molecular Mass}} \\ &= \frac{\dots\dots\dots \times 10}{287.36} = \dots\dots\dots\end{aligned}$$

(i) Standardisation of EDTA

$$\text{Volume of ZnSO}_4 \text{ solution, } V_1 = 20 \text{ ml}$$

$$\text{Volume of EDTA, } V_2 = \dots\dots\dots \text{ ml}$$

$$\begin{aligned}\text{Molarity of EDTA solution, } M_2 &= \frac{V_1 \times M_1}{V_2} \\ &= \dots\dots\dots\end{aligned}$$

(ii) Estimation of Magnesium

$$\text{Volume of Mg}^{2+} \text{ solution, } V_3 = 20 \text{ ml}$$

$$\text{Volume of EDTA, } V_4 = \dots\dots\dots \text{ mL}$$

$$\text{Molarity of EDTA solution, } M_2 = \dots\dots\dots$$

$$\begin{aligned}\text{Molarity of Mg}^{2+} \text{ solution, } M_3 &= \frac{V_4 \times M_2}{20} \\ &= \dots\dots\dots\end{aligned}$$

$$\begin{aligned}\text{Mass of Mg}^{2+} \text{ per litre of the given solution, } W &= M_3 \times 24.01 \\ &= \dots\dots\dots \text{ g}\end{aligned}$$

titrated against EDTA until the colour of the solution changes from wine red to blue (at the end point). The titration is repeated for concordant titre values.

3. Estimation of Magnesium

Given Mg^{2+} solution is made upto 100ml in distilled water. 20 ml of this solution is pipetted out into a conical flask. 2 ml of the buffer solution and 4 drops of Eriochrome black T indicator are added to this solution, and titrated against the standard EDTA until the colour changes from red to pure blue. Titrations are repeated till concordant results are obtained. From the titer values, strength of magnesium solution and the amount of magnesium in the whole of the given solution is calculated.

Result:

Mass of the magnesium present per litre of the given solution =g

Observation:

Weight of brass sample taken = _____ g

Sl. No.	Volume of Copper Solution (mL)	Concentration (mg)	Absorbance
1.	1	5	
2.	2	10	
3.	3	15	
4.	4	20	
5.	5	25	
6.	sample	?	

Plot a calibration curve on a graph paper and deduce the concentration of the Brass sample.

Concentration of Copper (in 5 mL) = _____ mg

Concentration of Copper (in 50 mL) = _____ mg = χ

% Cu = $\frac{\chi \text{ mg}}{\text{Wt. of brass in mg}} \times 100 = \text{_____ \%}$

Experiment No.....

Date

Colorimetric estimation of copper in brass

Objective:

To determine the amount of copper present in brass by colorimetric method

Theory:

Alloys primarily contain different metals in varying proportions. Properties of an alloy depend on their compositions. The knowledge of compositions is essential for quality control. The colored compounds find a place in quantitative analysis of materials as the intensity of the species is directly related to its concentration. The concept is used in the determination of the amount of copper present in the given sample of 'brass'.

Procedure:

Weigh out accurately about 0.12 g of brass and transfer it into a 100 mL conical flask. Add to this, 10 mL of 1:1 nitric acid and boil gently for 10 minutes until the evolution of brown gas ceases completely.



To the above solution, add 10 mL of water, one gram (one spatula) of urea and boil for few minutes. Cool the resultant solution to room temperature, transfer quantitatively into a 50 mL standard flask and make up to the mark. Pipette out 5 mL of this solution into a 25 mL standard flask containing 5 mL of 1:1 ammonia solution. The solution is mixed well and made up to 25 mL. Measure the absorbance of this solution using a 610 nm filter.

From the given stock solution, pipette out 1, 2, 3, 4 and 5 mL into 5 different 25 mL standard flasks and add 5 mL of 1:1 ammonia solution in each of them and make up to the mark. Mix well. Prepare a blank solution without CuSO_4 and measure the absorbance of each of this solution using the same filter. Tabulate the readings, draw a calibration graph and determine the copper content in brass using the graph.

Result:

The percentage of copper content present in the given brass sample is _____%

Observations:

Mass of solid A in g	Mass of solid B in g	Mass percentage of solid A	Mass percentage of solid B	Melting point in °C

1. The eutectic temperature of the mixture = °C
2. The composition of the binary mixture at eutectic point =
3. The composition of the given unknown mixture =

Experiment No.....

Date

Determination of Iron in Haematite Ore

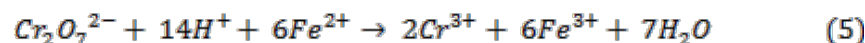
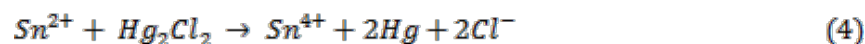
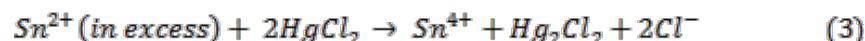
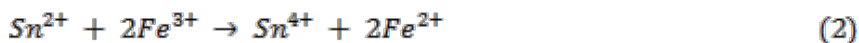
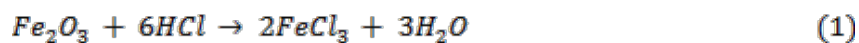
Objective:

To determine the percentage of Iron in Haematite ore.

Theory:

A known weight of the Haematite ore is dissolved in hot conc. HCl, and the ferric chloride produced is reduced to ferrous chloride using stannous chloride solution. The excess SnCl₂ is destroyed by adding mercuric chloride. For the estimation of Fe(II), the solution is now titrated against standard K₂Cr₂O₇ solution using 1% diphenylamine (DPA) in conc. H₂SO₄ as indicator. Orthophosphoric acid (H₃PO₄) is used to complex the ferric ion as [Fe(HPO₄)]⁺.

The E₀ value for the Fe(III)/Fe(II) system is +0.77 volt. Initially only traces of Fe(III) may be present and if the ratio (Fe³⁺/Fe²⁺) is taken as 10⁻⁴, E₀ is only +0.53 V and the indicator is colourless. However, when 50% of Fe(II) is oxidized to Fe(III), E₀ = 0.77 and so the indicator changes colour prematurely. In order to avoid this, the concentration of Fe(III), is decreased by complexing with phosphoric acid and thus the potential is kept below 0.77. However, very near the end point, since Fe²⁺ is extremely small, the value of E₀ increases sharply and the indicator changes its colour.



Equivalent weight of Fe₂O₃ is formula weight/2, and that of iron is Fe/1 or 55.85. Since the conversion of Fe(III) to Fe(II) involves a change in oxidation state of unity.

Procedure:

1. Dissolution of Haematite in Conc. HCl

To the given Haematite ore (0.4 to 0.5g) in the beaker add 15 to 20 ml of conc. HCl. Cover the beaker with a watch glass. Keep it inside a fume chamber and heat it gently (70-90°C) to digest the ore till the ore particles dissolve completely (30-40 min). Add sufficient conc. HCl to get a clear solution of ferric chloride. Cool, transfer the solution quantitatively into a 250 ml standard flask and make up to the mark.

Observation:

Weight of the Haematite (~0.5 g) = ----- g

Sl. No.	Vol. of Fe (II) solution pipetted out (V_2 ml)	Burette Reading		Volume of $K_2Cr_2O_7$ (V_1 ml)
		Initial	Final	
1				
2				
3				

Best titre value = -----ml (V_1)

Strength of $K_2Cr_2O_7$ solution = ----- (N_1)

Strength of Fe (II) solution = $(N_1 V_1)/V_2$ = ----- (N_2)

Weight of Fe(II) in the made up solution = $(N_2 \times 55.85)/4$ =----- g

Weight of the Haematite = ----- g

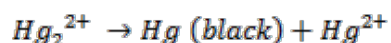
% of iron in Haematite = $\frac{\text{Wt. of iron in solution}}{\text{Wt. of ore}} \times 100$

= ----- %

2. Reduction of FeCl₃ into FeCl₂

Pipette out 25 ml of the made up solution into a conical flask, add 10 ml of conc. HCl and heat to boiling. Add SnCl₂ solution drop wise to the hot Fe(III) solution till the yellow colour due to ferric ion is just discharged. Cool rapidly under the tap, add 5 ml of 5% solution of aq. mercuric chloride (HgCl₂) in one lot quickly with thorough mixing to avoid the reaction (4). The reaction as given in equation (4) is caused by the local excess of the SnCl₂ in the solution. If the addition of HgCl₂ leads to a silky white precipitate, we can proceed with the experiment. If a black or grey precipitate is obtained, we have to discard the contents and a fresh solution has to be taken for reduction.

Note: The HgCl₂ solution must be added after cooling the reduced solution, otherwise, the following disproportionation reaction may set in



The whole procedure warrants repetition. The finely divided Hg formed will get oxidized by K₂Cr₂O₇ solution and also the visual detection of end point becomes rather difficult. Small quantities of Hg₂Cl₂ in suspension are not appreciably oxidized by dichromate.

3. Determination of Fe²⁺ got from Fe³⁺ of the ore (Fe₂O₃)

If a silky white precipitate is obtained on adding 5% HgCl₂ solution, add 2 ml of Orthophosphoric acid, 10 ml of 2 N HCl and 3 drops of DPA indicator. Titrate this solution against standard K₂Cr₂O₇ (~0.05N) solution until the solution turns bluish violet. This is the end point of this redox titration. Repeat the steps 2 and 3 with a fresh portion of the made up solution and calculate from the titre value, the percentage by weight of iron in the haematite sample.

Result:

The percentage of iron in haematite ore is = -----

Experiment No.....

Date

Phase rule: Two component systems

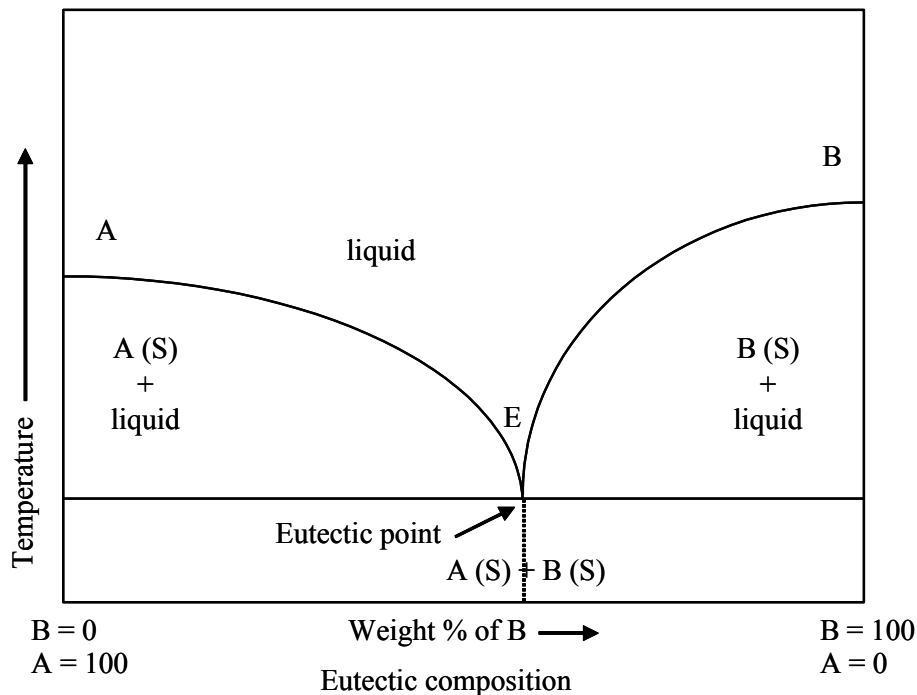
Objective:

To determine the eutectic temperature and composition of a two component (solid) system.

Theory:

A diagram which illustrates the conditions of equilibrium between various phase of a substance, is called a phase diagram or equilibrium diagram. These conditions of equilibrium between various phases are summarized in a theoretically deduced rule called Gibbs phase rule, which can be stated mathematically as

$$F = C - P + 2$$



In the study of solid-liquid condensed phase equilibria it is the practice to disregard the vapour phase and to fix an arbitrary constant pressure, generally 1 atmosphere (condensed system). The reduced form of the phase rule, namely, $F = C - P + 1$ is used to represent the conditions of equilibrium between various phases. If a liquid mixture of two similar components A and B is cooled, solid will commence to separate at a definite temperature, namely, the freezing point. The actual value of the freezing point will depend on the

Observations:

Mass of solid A in g	Mass of solid B in g	Mass percentage of solid A	Mass percentage of solid B	Melting point in °C

1. The eutectic temperature of the mixture = °C
2. The composition of the binary mixture at eutectic point =
3. The composition of the given unknown mixture =

composition of the liquid mixture, and if the results for series of such mixtures of components varying from pure A to pure B are plotted against the corresponding compositions, two curves like AE and BE (See figure) are obtained. The curves AE and BE may be regarded as representing the conditions of temperature under which liquid phase of various compositions are in equilibrium with the solid phase A or the solid phase B respectively. At the point E, where the curves, AE and BE meet, both the solids A and B are in equilibrium with the liquid phase. This means that there are three phases at the point E and since it is a two component system applying the reduced phase rule, the number of degrees of freedom $F = 2 - 3 + 1 = 0$. There is thus only one temperature at which any liquid mixture can be in equilibrium with both solids A and B. Consequently, it is the lowest temperature at which any mixture of solid A and B will melt. Hence, the point E is called the 'eutectic point' characterized by eutectic temperature and eutectic composition.

Procedure:

Weigh 5g of solid A accurately and transfer the solid in to a pyrex boiling tube which is provided with a glass stirrer and a thermometer. Immerse the test tube in a water bath and heat the water bath till the solid melts. Remove the boiling tube from the water bath and cool it in air with constant stirring. Find out the melting point of the solid A by noting the temperature at which solid A crystallizes out (note the first appearance of the solid) from the liquid phase. Weigh 1 g of solid B accurately and add this to the test tube containing 5g of A. Again melt the mixture by immersing the composition. Cool the test-tube in air and find out the melting point of the mixture. Now increase the amount of solid B to the contents of the same test tube. Find out the freezing point of the mixture, as before, for this composition. Increase the addition of B slowly by adding each time one gram till 50 % by weight of B is reached (i.e. 5g of A and 5g of B). Repeat the procedure by taking 5g of solid B and adding 1, 2, 3, 4 g of solid A and noting the freezing points of the mixture of various compositions each time. Tabulate your result in the record of observation. Plot temperature of the freezing mixture versus weight % of B. Report the eutectic temperature and the eutectic composition. Find the freezing point of the unknown mixture given. Add 1g of either A or B and find the new freezing point. From this determine the composition of the unknown mixture.

Result:

1. The eutectic temperature of the mixture = °C
2. The composition of the binary mixture at eutectic point =
3. The composition of the given unknown mixture =

Experiment No.....

Date

Analysis of organic compounds

Tests for functional groups

Experiment	Observation	Inference
Test for Acids: a) NaHCO₃ test: Sample + 1 mL of NaHCO ₃ solution b) NaOH test: 0.1g of substance (3 drops) + 1 mL 10 % NaOH solution. Shake well. All organic acids dissolve when shaken with NaOH.	 A brisk effervescence with the evolution of CO ₂ which turns lime water milky On acidifying with conc. HCl, the compound is precipitated.	
Test for Aldehydes and Ketones: a) Borsche's test: Substance + ~ 1 mL Borsche's reagent, boil and add Conc.HCl, cool, add 1 mL water. b) Fehling's Solution Test: (for Aliphatic Aldehydes only) Sample + ~ 1 mL each of Fehling's solution "A" & "B". Heat to boiling.	 Yellow or orange precipitate Red precipitate	

<p>c) Tollen's Reagent test: (Aldehyde only)</p> <p>Sample + ~ 1 mL Tollen's reagent, Heat the test tube in a water bath for 5-10 mins.</p> <p>d) Legal's test: (for Ketones only)</p> <p>Sample + 2 mL water + 5 drops of sodium nitroprusside solution + 5 drops NaOH + 5 drops NaOH + 5 drops CH₃COOH</p>	<p>Black precipitate or bright silver mirror</p> <p>Orange colour changes to purple</p>	
<p><u>Test for Phenol</u></p> <p>Sample + 1 mL of neutral Ferric chloride</p> <p><u>Test for Amines</u></p> <p><u>a) Solubility test:</u></p> <p>0.1g substance + ~1 mL dil HCl. Shake well.</p> <p>b) Dye Test:</p> <p>Sample + ~3 mL dil. HCl, cool (in ice), add 1 mL NaNO₂ solution and 1 mL alkaline -β-naphthol</p>	<p>A violet colour</p> <p>Substance is soluble and gets precipitated on adding NaOH</p> <p>A scarlet red dye</p>	
<p>Result:</p> <p>1. The given unknown sample is _____</p>		

Rough Work

Rough Work

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