MIT WORLD PEACE UNIVERSITY, PUNE

First Year B. Tech

School of Chemistry

Subject: Chemistry (Code: UCH1001A)

LABORATORY MANUAL

2021-22

Preface

Chemistry

The laboratory work included for the subject Chemistry for F.Y.B. Tech students of MIT-World Peace University, covers experiments which are of interest to students of engineering stream. The experiments based on Water, Coal, Corrosion and Polymer science includes the tests which are routinely carried out on these materials. The students will be familiar with the principles, significance and utility of various chemical analysis techniques and also will understand the precautions to be taken during experimentation of these techniques. In order to help students' probe, their grasp of the subject, questions based on each experiment have been included. The safety precautions required in the laboratory are also included in this manual. We hope that this course will provide hands on experience and lifelong learning to the students.

General Instructions

1. Follow your timetable strictly.
2. Always carry printed manual of experiment for scheduled practical with you.
3. Must wear the I-card
4. Always carry note book to record the results and to do calculations.
5. Wear the lab coat (white apron) during the practical.
6. Tie your hair and dupatta in laboratory.
7. Do not eat food, drink beverages or chew gum in laboratory.

Safety Instructions

Chemistry laboratory is associated with several potential hazards and therefore certain precautions should be taken to minimize the probability of an accident. The awareness of hazards is the most important factor to avoid accidents and hence safety in the laboratory.

1. Before you begin your work, see that all the requirements of the experiment are on the table. Perform the experiment in presence of teacher
2. Do not touch any equipment, chemicals or other materials unless you are told to do so.
3. Handle the apparatus very carefully.
4. Follow following precautions to avoid breakage.
   - Do not clamp retorts, flasks or other glass apparatus too tightly.
   - Always heat gradually and uniformly.
   - Before applying the flame confirm that apparatus is dry.
   - Take care to see that no water splashes in anyway on the hot apparatus.
5. Do not throw any rubbish (broken glass, filter paper, pieces of paper etc.) in the sink.
6. Be always particular to use the necessary amount of material to avoid wastage.
7. Turn on the water tap very slowly.
8. Take care while handling acids and place the stoppers of acid bottles on them after use.
9. Have only your note book on the table, and other things should be kept in the rack.
10. Before you leave the laboratory you should clean apparatus & keep it neatly on the table.
11. Record all observations in your notebook.
12. Report all accidents to your teacher immediately even if you think it is minor.
13. Put off the burner immediately after use.
14. In case of eye accident, wash the eye with water for at least 10 minutes.

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INDEX

Sr. No.	Name of the Experiment	Page	Date	Sign of Batch I/C
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2	To determine alkalinity of given water sample.			
3	Estimation of moisture and ash content in a given sample of coal.			
4	Demonstration of effect of environmental conditions on metal corrosion.			
5	To determine the electro chemical equivalent (ECE) of copper.			
6	To prepare Nylons and to draw them in the form of thread.			
7	Determination of iron concentration in a given sample using Colorimeter.			
8	Study of the adsorption of metal ions on plant-based adsorbents using UV-visible spectrophotometer.			
9	Determination of the molecular weight of a polymer by using Ostwald's Viscometer.			
10	Estimation of dissolved oxygen in the given samples of water by Winkler's method.			

CERTIFICATE

Certified that Mr. /Ms. _____ of F.Y.B. Tech. Division _____ Roll No._____ has completed the laboratory work in the subject **Chemistry** During the trimester-I/II/III of the academic year_____

Signature of the Faculty **Seal of the Head of the Department**

Name: _____. **Class:**_____. **Batch:**_____.

Roll No.: _____ **Experiment No: 01**

Performed on: _____. **Submitted on:**_____. **Teacher's Sign :** _____.

Aim: To estimate total hardness of water by EDTA method.

Objective:

To determine the hardness of a water sample by complexometric titration method. Ethylene-diamine-tetraacetic acid (EDTA) is used as chelating agent. It forms complex with divalent cations such as Ca^{2+} and Mg^{2+} ions in stoichiometric amount and thus hardness can be determined as ppm of $CaCO_3$.

Apparatus:

Burette, Conical Flask, 100ml volumetric Flask, Beaker, Watch glass, Burette Stand etc.

Chemicals:

Approx. 0.01 M Na_2EDTA, 0.01M $ZnSO_4$, Buffer Solution (pH=10), Hard water Sample, Eriochrome Black-T [EBT] etc.

Theory:

Water sample which does not produce lather readily with soap solution is known as hard water. On heating, it leaves deposits of scales on the walls of the container. The total hardness is due to the presence of dissolved bicarbonates, sulphates, chlorides and nitrates of magnesium calcium and other divalent cations of metal.

The relation between the type of water sample and degree of hardness can be given by following table.

Hardness description of water sample	Hardness as ppm of $CaCO_3$
Soft	0-75
Moderately hard	75-150
Hard	150-300
Very hard	Above300

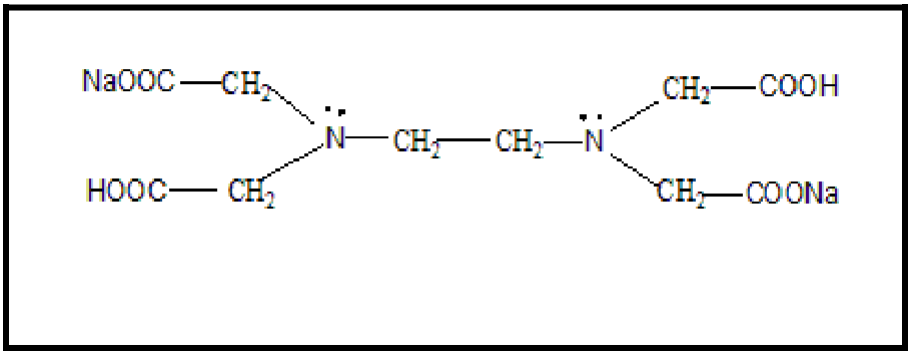
The hardness which can be easily removed on heating the water sample is known as temporary hardness. It is due to bicarbonates of calcium and magnesium which get decomposed on heating as insoluble carbonates and hydroxides which can be removed by filtration.

$Ca(HCO_3)_2 \longrightarrow CaCO_3\downarrow + H_2O + CO_2\uparrow$

$Mg(HCO_3)_2 \longrightarrow Mg(OH)_2\downarrow + 2CO_2\uparrow$

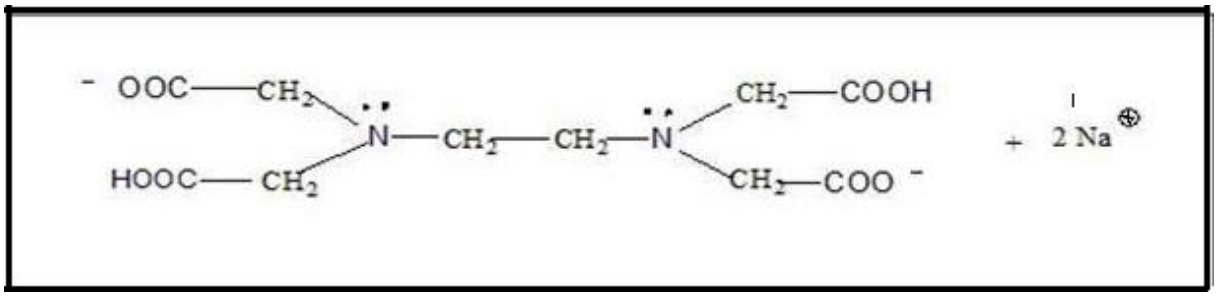
This hardness is also known as bicarbonate hardness. The hardness which cannot be removed by heating the water sample is known as permanent hardness or non-carbonate hardness. The sum of temporary and permanent hardness is known as total hardness.

For determining suitability of water for domestic and industrial purpose, type of hardness and hence magnitude of hardness is important. To determine total hardness of given water sample, disodium salt of ethylene diamine tetra acetic acid is used as it forms strong 1:1 complex with divalent metal ions.



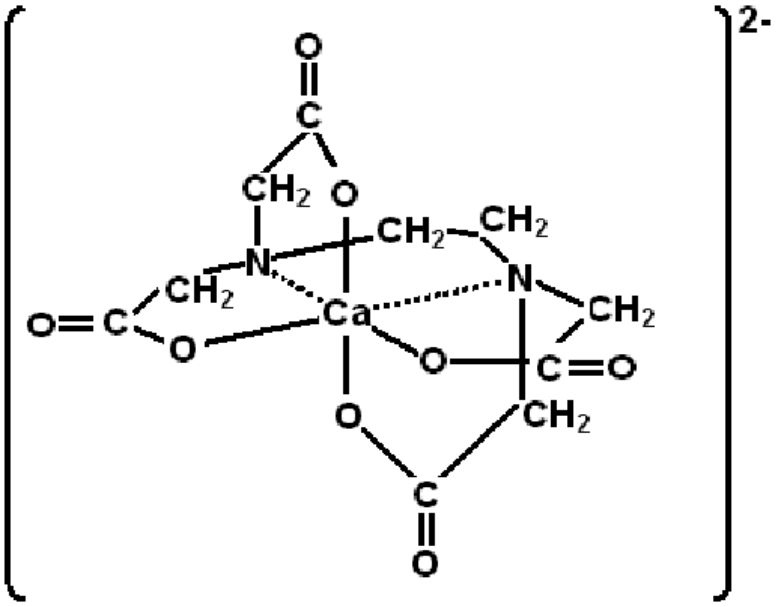
Di-sodium salt of EDTA

The disodium salt of EDTA ionizes in water as;



This anion is a strong chelating agent and can be represented as H_2Y^{2-}

The Na_2EDTA forms a stable complex in basic medium, thus the alkaline buffer of NH_4OH and NH_4Cl of pH=10 is used.



Molecular structure of Ca-EDTA complex

In this Complexometric titration, EBT (Eriochrome Black-T) is used as an indicator. It forms less stable wine red colored complex with metal ions. (Less stable w.r.t. metal ion– EDTA complex). But when Na_2EDTA is added to it, metal-indicator complex dissociates setting metal ions free. They immediately form stable colorless complex with Na_2EDTA. As a result, solution appears blue, which is the color of free indicator ions. Appearance of blue color is taken as end point of titration.

$$M^{2+} + HIn^{2-} \xrightarrow{pH = 10} MIn^{-} + H^{+}$$

Indicator — Metal indicator complex

(blue) — (wine red)

$$MIn^{-} + H_2Y^{2-} \xrightarrow{pH = 10} MY^{2-} + HIn^{2-} + H^{+}$$

MY^{2-}: Metal– EDTA Complex (colourless)

HIn^{2-}: Indictor (blue)

Procedure:

Part – A:- Preparation of standard 0.01 M $ZnSO_4.7H_2O$

Weigh accurately 0.2872g of pure $ZnSO_4$ on a watch glass. Transfer it to 250 ml beaker. Dissolve it in distilled water. Take washings of watch glass. Transfer the solution to 100 ml volumetric flask. Take washings of beaker also. Dilute the solution up to the mark with distilled water and shake it to make it homogeneous.

Part – B: -Standardization of Na_2EDTA solution by double burette method

Fill the burette one with given Na_2EDTA (approx.0.01M) solution. Fill burette two with above standard $ZnSO_4$ solution. Take by burette two, 5ml of 0.01M standard solution of $ZnSO_4$ in conical flask. Add 3ml of buffer of pH10 and 3 drops of Eriochrome black– T indicator. Titrate it against Na_2EDTA till color changes from wine red to blue. Let this reading be X_1 ml.

To the same flask add one ml (6ml) of std. $ZnSO_4$, by burette. Add Na_2EDTA solution by burette one till blue colour appears. Let this reading be X_2 ml.

To the same solution add one ml (7ml) of $ZnSO_4$ solution by burette two. Add Na_2EDTA solution from burette one till blue colour appears. Let this reading be X_3 ml. From these three readings calculate exact molarity of Na_2EDTA solution.

Precautions

1. Do not inhale/pipette out buffer solution as it contains ammonia solution and it causes irritation if inhaled.
2. Do not use buffer solution if it is turbid.

Part – B :-Observation table

Burette1 - Na_2EDTA solution (0.01 M) approx

Burette2 - $ZnSO_4$ solution (0.01 M)

Indicator - Eriochrome Black-T

End point - Wine red to blue

Reaction - $Na_2EDTA + Zn^{2+} \longrightarrow [EDTA\ (Zn^{2+})\ complex]^{2-} + 2\ Na^{+}$

Burette 1	Na_2EDTA	X_1=	X_2=	X_3=
Burette 2	$ZnSO_4$	5 ml	6 ml	7 ml

Calculation To calculate exact molarity of EDTA (0.01M approx.)

$M1 \times V1 = M2 \times V2$

$EDTA \equiv ZnSO_4$

$M1 \times X1 = 0.01 \times 5$

$$M_1 = \frac{0.01 \times 5}{X1}$$

$M_2 \times X_2 = 0.01 \times 6$

$$M_2 = \frac{0.01 \times 6}{X2}$$

$M_3 \times X_3 = 0.01 \times 7$

$$M_3 = \frac{0.01 \times 7}{X3}$$

Molarity of EDTA $M' = \frac{M1 + M2 + M3}{3}$

Exact Molarity of Na_2EDTA = M' =..... Molar

Part – C:-To find out total hardness of given water sample

Fill the burette one with given Na_2EDTA (approx.0.01M) solution. Fill burette two with given hard water sample. Take by burette two, 5ml of hard water in a conical flask. Add 3ml of buffer of pH 10 and 3 drops of Eriochrome black–T indicator. Titrate it against Na_2EDTA till colour changes from wine red to blue. Let this reading be Y_1 ml.

To the same flask add one ml (6ml) of hard water by burette two. Add Na_2EDTA solution by burette one till blue colour appears. Let this reading be Y_2 ml.

To the same solution add 1ml (7ml) of hard water by burette two. Add Na_2EDTA solution from burette one till blue colour appears. Let this reading be Y_3 ml.

Observation table

Burette1	-	Na_2EDTA solution (M')
Burette2	-	Hard water sample
Indicator	-	Eriochrome Black-T
End point	-	Wine red to blue

Equations:

$HIn^{2-} + Ca^{++} \longrightarrow CaIn^- + H^+$

(Indicator) blue — Wine red coloured complex

$CaIn^- + H_2Y^{2-} \longrightarrow CaY^{2-} + HIn^{2-} + H^+$

Wine red — Colourless Complex — blue (Indicator)

Burette 1	Na_2EDTA	Y_1=	Y_2=	Y_3=
Burette 2	Hard water sample	5 ml	6 ml	7 ml

Calculations: -

EDTA and Ca^{2+} (or Mg^{2+}) ions form 1:1 complex.

1 mole of EDTA ≡ 1 mole of Ca^{2+} (or Mg^{2+}) ≡ 1 mole of $CaCO_3$
1 mole of EDTA ≡ 100g $CaCO_3$
Thus 1000 ml1 M EDTA ≡ 100 g of $CaCO_3$
1 ml 0.01 M EDTA ≡ 1 mg of $CaCO_3$

$$Y_1 ml\ M'\ M\ Na_2EDTA = \frac{Y_1 \times M'}{0.01}$$

$= A_1$ mg of $CaCO_3$

As 5ml of water sample contains A_1 mg of $CaCO_3$.
1000 ml of water sample contains 200 A_1 mg of $CaCO_3 = H_1$
But 1 mg of $CaCO_3$ per liter (10^6mg) of water is 1 part per million of $CaCO_3$ (1ppm).
Thus hardness of water sample is 200 A_1 ppm.

For Y_2ml,

$$Y_2 ml\ M'\ M\ Na_2EDTA = \frac{Y_2 \times M'}{0.01}$$

$= A_2$ mg of $CaCO_3$

1000 ml of water sample contains 167 A_2 mg of $CaCO_3 = H_2$
But 1 mg of $CaCO_3$ per liter (10^6mg) of water is 1 part per million of $CaCO_3$ (1ppm).
Thus, hardness of water sample is 167 A_2 ppm.

For Y_3ml,

$$Y_3 ml\ M'\ M\ Na_2EDTA = \frac{Y_3 \times M'}{0.01}$$

$= A_3$ mg of $CaCO_3$

As 7 ml of water sample contains A_3 mg of $CaCO_3$
1000 ml of water sample contains 143 A_3 mg of $CaCO_3 = H_3$
But 1 mg of $CaCO_3$ per liter (10^6mg) of water is 1 part per million of $CaCO_3$ (1ppm).
Thus hardness of water sample is 143 A_3 ppm.

$$\text{Then hardness of given water sample} = \frac{H_1 + H_2 + H_3}{3} = \cdots \ldots \ldots \ldots ppm\ of\ CaCO_3$$

Results:

1. **Exact molarity of Na_2EDTA solution =… ..... molar**
2. **Total hardness of a given sample of water = ... ppm of $CaCO_3$**

Conclusion:

Questions:

1) Explain the significance of determination of hardness of water.

2) Why the end point of titration is wine red to blue?

3) Why and how is the pH value adjusted to about 10?

4) As per WHO norms, what is the standard value of hardness for drinking water?

5) What is potable water, deionized water, saline water, brackish and mineral water?

Note: Students are instructed to do all necessary calculations and answer the questions on separate sheets and attach them.

Dr. Vishwanath Karad MIT World Peace University

F.Y. B. Tech. Academic Year 2021-22 Trimester:

SCIENCE & ENGG. LABORATORY CONTINUOUS ASSESSMENT

DIMENSION	SCALE					SCORE
	1	2	3	4	5	
Regularity and punctuality	Did not Perform /submit	Performed and Submitted later than scheduled date with permission	Performed on schedule; Submitted two weeks late	Performed on schedule; Submitted one week late	Performed and submitted As per schedule	
Understanding The Objective	Neither shows any Understanding of the objective nor can relate it to theory	States the objective Very vaguely	Can only state the Objective but shows poor understanding	Understands objective But can not place it in context of a theory topic	Understands objective and Can relate it to an appropriate theory topic	
Understanding of Procedure	Cannot follow the Procedure and do any work	Follows the procedure half-heartedly	Follows right procedure; But cannot analyze data and interpret it	Follows right procedure, can analyze data but cannot interpret it	Follows right procedure, Can analyze data and interpret it with justification	
Experiment Skills	Does not participate in The experiment	Performs the Experiment only with the help from supervisor/others and Is confused and untidy.	Performs the experiment With some supervisory help, forgets some crucial readings. Is confused and untidy.	Performs experiment on Own without supervisor's help; records all the readings properly but is untidy.	Performs experiment on Own without supervisor's help and records all the readings properly. Keeps the set-up clean and tidy.	
Ethics	Copies the results from others	Completes the result analysis with help from others but forgets to acknowledge the help.	Completes the result analysis with help from others and acknowledges the help.	Produces his own result Analysis but blames others for any inadequacy found during the examination	Produces his own result Analysis faithfully and owns up the results without any manipulation	
Total =						

Teacher's Signature with Date: **Student's Signature with Date:**

Name: _____. **Class:** _____. **Batch:** _____.

Roll No.: _____ **Experiment No: 02**

Performed on: _____. **Submitted on:** _____. **Teacher's Sign.:** _____.

Aim: To determine alkalinity of given water sample.

Objective

To determine the alkalinity i.e. ability of water to maintain constant pH due to carbonate, bicarbonates and hydroxide ions present in water. The alkalinity of water is determined by titrating sample against standard solution of acid is an acid-base titration.

Apparatus:

Burette, Pipette, Conical flask (250 ml), Volumetric Flask, Burette Stand etc.

Chemicals:

0.02N HCl, Phenolphthalein indicator, Methyl Orange indicator, water Samples etc.

Theory:

Alkalinity is the way of measuring acid neutralizing capacity of water. In other way, it is ability of water to maintain constant pH. The alkalinity of water is due to the presence of hydroxide ion (OH^-), carbonate ion (CO_3^{2-}) and bicarbonate ion (HCO_3^-) present in the given sample of water.

These can be estimated separately by titration of the water sample against standard acid using phenolphthalein and methyl orange as indicators. The chemistry involved can be shown by the equations given below;

i) $H^+ + OH^- \longrightarrow H_2O$

ii) $H^+ + CO_3^{2-} \longrightarrow HCO_3^-$

iii) $HCO_3^- + H^+ \longrightarrow H_2O + CO_2$

The titration of the water sample against a standard acid up to the phenolphthalein end – point shows the completion of reactions (i) and (ii) only. The volume of the acid (V_1ml) used up to this point thus corresponds to complete neutralization of hydroxide and conversion of all the carbonate to

bicarbonate. The alkalinity measured till this point is called Phenolphthalein Alkalinity. The end point is called phenolphthalein end point.

The titration of the water sample is continued using methyl orange indicator which causes colour change from yellow to red at a pH of about 4.5. The additional volume of acid (V_2ml) corresponds to complete neutralization of bicarbonate ions (see reaction iii). The end point is called methyl-orange end point.

The possible combinations of ions causing alkalinity in water are;

(i) OH^-only, CO_3^{2-}only, HCO_3^-only or

(ii) $OH^-$and CO_3^{2-}, or $CO_3^{2-}$and HCO_3^-together

(iii) The possibility of OH^- and HCO_3^- ions together is not possible since they combine to form CO_3^{2-}ions.

$$\mathbf{OH^- + HCO_3^- \longrightarrow CO_3^{2-} + H_2O}$$

The total volume of acid (V_2) ml corresponds to the neutralization of hydroxide, carbonate and bicarbonate and hence gives the measure of total alkalinity or methyl orange alkalinity and can be expressed as parts of equivalent $CaCO_3$ per million parts of water.

Procedure:

First check the pH of the given sample of water with pH paper. Pipette out 100ml of water sample in conical flask. If pH of the sample is above 8.5 units, then titrate it against 0.02N HCl using phenolphthalein as an indicator, till pink color just vanishes. Let this reading be V_1 ml. To this solution add 3 to 4 drops of methyl orange as an indicator and continue the titration till yellow color changes to red. Let this reading be V_2 ml from the beginning. This is called as methyl orange end point.

This procedure is repeated for various water samples.

Precautions:

1. To avoid loss of CO_2, the titration should be carried out quickly and vigorous shaking should be avoided.
2. Add phenolphthalein indicator only if the pH of the water sample is above 8.5 units

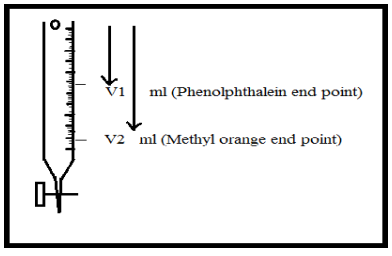


Figure: Titration of alkaline water sample against 0.02 N HCl solution

Observations:

Burette 0.02N HCl Solution

Pipette Water sample

Indicators
1. Phenolphthalein
2. Methyl orange to the same solution after getting first end point

End point
1. Pink to colourless
2. Yellow to red

Observation Table:

Sample No.	Readings in ml							
	V_1				V_2			
	I	II	III	Constant	I	II	III	constant
1								
2								
3								

Standard Reference Table: Type of alkalinities present in water samples

Relation between V_1 and V_2	Relation between P and M	Alkalinities in ppm		
		Hydroxide (OH^-)	Carbonate (CO_3^{2-})	Bicarbonate (HCO_3^-)
$V_1 = 0$	P=0	-	-	M
$V_1 = V_2$	P=M	M	-	-
$V_1 = ½V_2$	P=½ M	-	2 P	-
$V_1 > ½ V_2$	P>1/2 M	(2P -M)	2(M–P)	-
$V_1 < ½V_2$	P<½ M	-	2 P	(M – 2P)

Calculations:

Similar to hardness, alkalinity is also expressed as parts per million in terms of $CaCO_3$.

As 1000 ml of 1N HCl ≡ 50 g of $CaCO_3$

∴ 1 ml 1N HCl ≡ 50 mg of $CaCO_3$

V ml of 0.02 N HCl ≡ 50 x V x 0.02 = V mg of $CaCO_3$

This corresponds of 100ml of water sample.

∴ For 1000 ml water sample (10^6mg of water), alkalinity will be 10 V mg i.e. 10 V ppm of $CaCO_3$

Then phenolphthalein alkalinity P = 10 V_1 ppm

Methyl orange alkalinity M = 10 V_2 ppm

(1) For water sample having V_1 = 0, the water contains only bicarbonate alkalinity.

Methyl orange end point V_2 = ml

Methyl orange alkalinity M =10 V_2 = ... ppm

Alkalinity due to HCO_3^- = M = ... ppm

(2) For water sample having only hydroxide alkalinity, it gets neutralized at Phenolphthalein end point i.e. when V_1 ml acid is added. Here V_2 will be zero. Phenolphthalein end point

V_1 = …… ml

Phenolphthalein alkalinity P = 10 V_1 = ... ppm

Alkalinity due to OH^- = P = ... ppm

(3) If water sample contains only carbonate alkalinity, then Phenolphthalein end point corresponds to half neutralization of carbonate, then V_1 = ½ V_2

∴ Alkalinity due to CO_3^{2-} = 2P = ... ppm

(4) If water sample contains hydroxide and carbonate alkalinity, then, V_1 > ½ V_2

Phenolphthalein alkalinity = P = 10 V_1 = ... ppm

Methyl orange alkalinity = M = 10 V_2 = ... ppm

Alkalinity due to OH^- = (2P – M) = ... ppm

Alkalinity due to CO_3^{2-} = 2 (M – P) = ... ppm

(5) If water sample contains carbonate and bicarbonate alkalinity, then, $V_1 < \frac{1}{2} V_2$

Phenolphthalein alkalinity = P = 10 V_1 =..... ppm

Methyl orange alkalinity = M = 10 V_2 =..... ppm

∴ Alkalinity due to HCO_3^- = (M -2 P) =..... ppm

Alkalinity due to CO_3^{2-} = 2P =..... ppm

Results: -

Water Sample	OH^- , alkalinity in ppm	CO_3^{2-}, alkalinity in ppm	HCO_3^-, alkalinity in ppm
1			
2			
3			

Conclusion:

Questions:

1. What are the adverse effects of acidic and alkaline water?
2. Explain the significance of alkalinity determination.
3. What is the effect of temperature on the determination of alkalinity?
4. Name various ions which are responsible for alkalinity of water.
5. Alkalinity of water cannot be due to the simultaneous presence of OH^-, CO_3^{-2} and HCO_3^-. Give reason.

Note: Students are instructed to do all necessary calculations and answer the questions on separate sheets and attach them.

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Total =						

Teacher's Signature with Date: **Student's Signature with Date:**

Name: _____. **Class:** _____. **Batch:** _____.

Roll No.: _____ **Experiment No: 03**

Performed on: _____. **Submitted on:** _____. **Teacher's Sign.:** _____.

Aim: Estimation of moisture and ash content in a given sample of coal.

Objective:

Crucible, Desiccator, Pair of tongs, Electric Oven, Muffle furnace, weighing balance etc.

Chemicals:

Coal sample, anhydrous calcium chloride etc.

Theory:

To ascertain the quality of coal, proximate and ultimate analysis of coal is done. Proximate analysis is an empirical analysis which is essential to assess suitability percentage of coal for a particular application. Proximate analysis is determination of moisture content, volatile matter, ash content and fixed carbon content. This gives information about the practical utility of coal. Moisture content is the loss in weight of coal when heated in crucible at 110℃ for one hour.

The amount of volatile matter evolved and its composition depend on quality of the coal and also on temperature, rate of heating and time of heating. Ash content of coal is the weight of residue left after complete combustion of the coal sample at 750℃. Ash also reduces the quality of the coal. The percentage of fixed carbon is then calculated by summing up of % moisture, % volatile matter and % ash and subtracting it from 100.

Procedure:

Part -A: Moisture Content

Take approx. 1g (0.8gm to 1.2gm) of powdered coal sample in pre-weighed silica crucible. Heat the sample in an electric oven at 110℃ for about one hour. Take out the crucible and keep in desiccator to cool it to room temperature and then weight it. Note the weight of the crucible after you get constant weight.

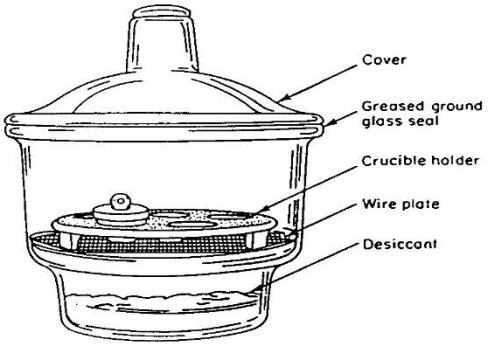

Figure :- Dessicator

Part -B: Ash Content

Take approx. 1g (0.8gm to 1.2gm) of powdered coal sample in pre-weighed silica crucible. Heat the coal without the lid in a muffle furnace at 750℃ for 30 minutes. Take out the crucible and cool and weigh it. The residue remaining in the crucible is the ash.

Precautions

a. The silica crucible should be weighed after heating and cooling till a constant weight is achieved.
b. The crucible or sample should be first cooled in desiccator before weighing.
c. The tongs should be used for putting and removing the crucible from Muffle furnace.

Part -A: Moisture Content

Sr. No.	Description	Value(g)
1	Weight of empty crucible **(w_1)**	
2	Weight of crucible and coal(w_2)	
	Weight of coal (w_2-w_1)	
3	Weight of crucible and coal after heating (w_3)	
4	weight of coal after heating(w_3-w_1)	
	Loss in weight (w_2–w_3)	

$$\%\ \textbf{Moisture} = \frac{\text{loss in weight}}{\text{weight of coal}}\ \text{X}\ 100 = \frac{\text{W2} - \text{W3}}{\text{W2} - \text{W1}}\ \text{X}\ 100$$

Part -B: Ash content

Sr. No.	Description	Value(g)
1	Weight of empty crucible **(w_1)**	
2	Weight of crucible and coal(w_2)	
	Weight of coal (w_2-w_1)	
3	Weight of crucible + ash after heating (w_3)	
4	weight of coal after heating(w_3-w_1)	
	Loss in weight (w_3–w_1)	

$$\% \text{ Ash } = \frac{\text{wt of ash formed}}{\text{wt. of dry coal taken}} \text{ X } 100$$

$$\% \text{ Ash } = \frac{W3 - W1}{W2 - W1} \text{ X}100$$

Result:

i) **% Moisture =……**

ii) **% Ash = ……..**

Conclusion:

Questions:

1. Explain the significance of moisture and ash content determination in coal?
2. What is the chemical composition of ash?
3. What is the difference between free ash and fixed ash?
4. What is inherent moisture present in coal?
5. At what temperature, moisture and ash present in coal are removed?

Note: Students are instructed to do all necessary calculations and answer the questions on separate sheets and attach them.

Dr. Vishwanath Karad MIT World Peace University

F.Y. B. Tech. **Academic Year:2021-22** **Trimester:**

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Teacher's Signature with Date: **Student's Signature with Date:**

Name: _____ **Class:** _____ **Batch:** _____

Roll No.: _____ **Experiment No: 04**

Performed on: _____ **Submitted on:** _____ **Teacher's Sign:** _____

Aim: Demonstration of effect of environmental conditions on metal corrosion

Objective:

To understand the effect of acidic environment of different concentration on corrosion of metal.

Apparatus:

50 ml Beakers, metal plates

Chemicals:

Sulphuric acid

Theory:

Corrosion is defined as the destruction of a solid body through chemical or electrochemical action starting at its surface. If a piece of metal is immersed in a polar solvent like water, some of metal ions leave the metal surface and go into the solvent or solution. As metal continues to dissolve, more and more electrons are left back and a net negative charge is built up in the metal. The potential developed can be measured under standard conditions using standard hydrogen electrode as a reference electrode. It is called standard reduction potential of metal. The metals having positive values are noble metals, they do not dissolve easily. (e.g. Au = +1.5 V, Cu = +0.522 V). The metals having negative values of electrode potential are called active metals, they go in the solution very easily (e.g. Fe = -0.044 V, Zn = -0.761 V).

The corrosion behavior of metals depends upon the environmental conditions. It can be easily studied by immersing metal plates in acid solutions of various concentration for a fixed or constant time.

Procedure: -

Take 3 metal plates (mild steel or zinc) of the 6 cm x 2.05 cm dimension. Rub the metallic plates with sand paper to remove any corrosion product on it. Weigh them separately and note down their weights. Prepare acid solutions having different concentration (1N, 2N and 4N). Take three 50 ml beakers and label them. Take 30 ml of the above prepared solutions in the beakers using measuring cylinder.

Insert the previously weighed metallic strips in the beaker so that 4/5 portion is immersed in the solution. Note down the time. After one hour take out the metallic plates from the beaker, dry them in air and then weigh. Note down their weights.

Observations:

Plate No.	Normality of the solution	Weight of the plate		Loss in weight	Loss / g / hr
		Before	After		
1	1 N				
2	2 N				
3	4 N				

Result:

i) Weight loss/g/h is maximum in the solution having conc. = …….

ii) Weight loss/g/h minimum in the solution having conc. = ……..…….

Conclusion:

Questions:

1. A solution is made up to contain 0.01 M HCl. What is its pH?
2. A solution is made up to contain 0.01 M NaOH. What is its pH?
3. A pure metal rod half immersed vertically in water starts corrosion at bottom. Justify.
4. What is the effect of temperature on the rate of wet corrosion?
5. Whether there will be corrosion in alkaline and neutral medium? Justify.

Note: Students are instructed to do all necessary calculation sand answer the questions on separate sheets and attach them

Dr. Vishwanath Karad MIT World Peace University

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Teacher's Signature with Date: **Student's Signature with Date:**

Name: _____. **Class:** _____. **Batch:** _____.

Roll No.: _____ **Experiment No: 05**

Performed on: _____. **Submitted on:** _____. **Teacher's Sign.:** _____.

Aim: To determine the electro chemical equivalent (ECE) of copper.

Objective:

To determine electrochemical equivalent of copper using copper voltameter, which is an electrolytic cell and the measurement is made by weighing the copper deposited at the cathode in a specified time.

Apparatus:

Copper plates, Voltameter, Ammeter, Rheostat etc.

Chemicals:

15% $CuSO_4$, 5% H_2SO_4 etc.

Theory:

Electrochemical equivalent is the mass of a substance liberated or deposited at an electrode during electrolysis is directly proportional to the quantity of charge passed through the electrolyte Faraday's First law of electrolysis states that "the weight of a substance deposited on an electrode during electrolysis is directly proportional to the quantity of electricity passed through the electrolyte".

If 'W' grams be the weight of substance deposited and 'Q' be the quantity of electricity in coulombs passed through an electrolyte,

Then,

$W \alpha Q$

But $Q = I \times t$ $\quad W \alpha I \times t$

$W = z.\ I.t$

$z = W / I \times t$

Where Q = number of coulombs
I = current in amperes
t = time in seconds
z = a constant known as ECE.

Copper Voltameter consist of copper anode and cathode and the solution is copper sulfate, acidified with sulfuric acid.

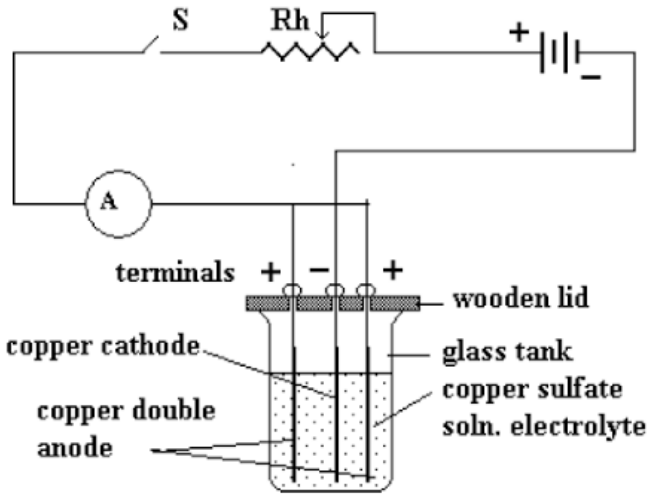

Figure: Copper voltammeter

Procedure:

Clean the copper cathode plate, weigh and connect it in the circuit as shown in figure. Take electrolyte solution (15% $CuSO_4$ + 5% H_2SO_4) in a copper voltammeter and dip the electrode plates in it. Then start the current and stopwatch simultaneously. Pass the current for about 45 min. Note down the reading of ammeter, when the electric current remains steady. Switch off the current and note the time for which current is passed (about 45 min). Remove the cathode plate, wash with distilled water, dry and weigh it accurately.

Precautions:

Hold the plate from its upper end so as to avoid touching its surface.

Cathode plate should be washed immediately after it is taken out from the solution of electrolyte.

The cathode plate should be dried completely before weighing.

Observation Table:

Sr. No.	Description	
1	Weight of cathode plate before deposition	W_1 = g
2	Weight of cathode plate after deposition	W_2 = g
3	Weight of cathode deposited (W_2-W_1)	W = g
4	Current passed in ampere	I =……. ampere
5	Time for which the current is passed	t = ……. seconds

Calculation:

Electrochemical equivalent (ECE) of Copper

$$z = \frac{w}{It} = \cdots \ldots \ldots \ldots g/c$$

Result:

Electrochemical equivalent (ECE) of Copper = …… g/C.

Conclusion:

Questions:

1. Define electrochemical equivalent (ECE)
2. Explain the significance of electrochemical equivalent (ECE) determination?
3. What is the effect of temperature on the determination?
4. What do you understand from the values of electrochemical equivalent (ECE) of following elements?

Element	**Electrochemical equivalent**
Silver	0.0011181
Copper	0.0003281
Hydrogen	0.0000104

5. Name and state the law which forms the basis of ECE.

Note: Students are instructed to do all necessary calculations and answer the questions on separate sheets and attach them.

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Teacher's Signature with Date: **Student's Signature with Date:**

Name:_____. Class:_____. Batch:_____.

Roll No.:_____ Experiment No: 06

Performed on:_____. Submitted on:_____. Teacher's Sign.:_____.

Aim: To prepare Nylons and to draw them in the form of thread.

Objective:

To prepare the nylons by interfacial polymerization technique in which two monomers are mixed in immiscible solvents and the polymerization takes place at the interface. The polymer film formed at interface is insoluble in both the solvents and can be drawn out in the form of a thread or a rope.

Apparatus:

250 ml Beakers, glass rod or test tube,

Chemicals:

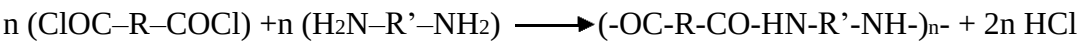
Sebacoyl chloride, Hexamethylene diamine, CCl_4, Distilled water, Methanol etc.

Theory:

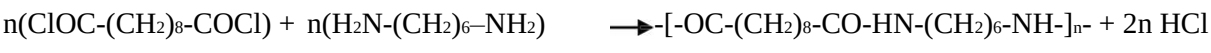
Nylons are prepared by interfacial polymerization technique. Interfacial polymerization is a type of step-growth polymerization in which polymerization occurs at the interface between an aqueous solution containing one monomer and an organic solution containing a second monomer. When aqueous solution of the diamine is carefully brought in contact with solution of di acid chloride in an organic solvent (immiscible with water), the reactant diffuse to the interface where the poly condensation reaction takes place. Since the polymer formed is at the interface of the liquid phases, the process is known as interfacial polycondensation. The solvents are selected such that the resulting polymer is not soluble in any of the solvent. The polymerization reactions are carried out between the temperature ranges 0 to 50^0C. In order to increase the conversion, the reaction mixture can be stirred. Due to stirring, the total area of reacting interface increases which in turn increases overall percent conversion. An inorganic base must be present in the aqueous phase to neutralize the byproduct HCl.

During polymerization, the polymer is formed at an interface, and the reaction between amine-chloride is very rapid. Therefore, polymer formed should be removed so that formation of a new polymer at the interface will be facilitated. This kind of technology is referred as to Nylon Rope Trick Method.

Reactions:



Diacid chloride **Diamine** **Polyamide**



Sebacoyl chloride **Hexamethylenediamine Nylon-6, 10**

Procedure:

Dissolve 1ml of sebacoyl chloride in 50 ml distilled CCl_4 in a 250 ml beaker. In another 250 ml beaker dissolve 2.2g of hexamethylene diamine in 50 ml of distilled water. The diamine solutions carefully poured in to the acid chloride solution along the sides of beaker so that it forms separate layer over the heavier CCl_4 solution layer. Grasp the polymer film formed at interfaces with tweezers and lift out of the beaker. As it comes out in the form of thread or rope, wrap it around a thick glass rod or test tube.

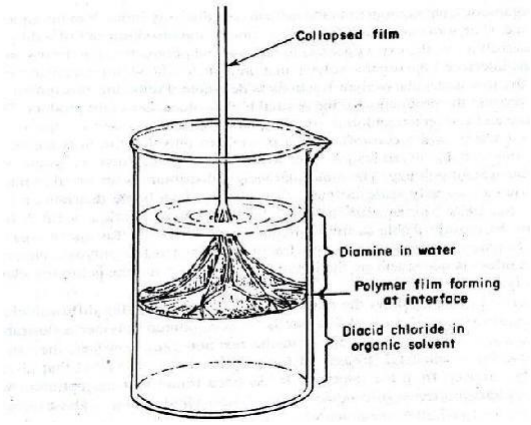


Figure - Interfacial Polymerization of diamine and diacid chloride giving nylon

Precautions:

1. Hexa methylene di amine is corrosive and irritates the skin so care should be taken to avoid its contact with skin.
2. The addition of diamine solution must be very slow and along the sides of beaker so that the lower layer of acid chloride solution is not disturbed.

Observation:

1. Nylon 6, 10 thread/rope is formed.
2. The two monomers soluble in solvent react to given Insoluble polymer– Nylon.
3. Reaction of 1ml of sebacoyl chloride with 2.2g of hexamethylene diamine yields…....g of Nylon.

Result:

Conclusion:

Questions:

1. Explain the terms - Addition and Condensation Polymerization.
2. What is Kevlar? Name the monomers used for its preparation.
3. Name the polymers which can be synthesized by interfacial polymerization technique.
4. Why thread or /rope of nylons can be withdrawn from the reaction mixture in interfaced polymer.
5. Is stoichiometric balance important for the success of interfacial polymerization? Why?

Note: Students are instructed to do all necessary calculations and answer the questions on separate sheets and attach them.

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Teacher's Signature with Date: **Student's Signature with Date:**

Name:_____. Class:_____. Batch:_____.

Roll No.: _____ **Experiment No: 07**

Performed on: _____**. Submitted on:** _____**. Teacher's Sign.:** _____.

Aim: To determine iron concentration in a given sample of water using Colorimeter.

Objectives:

To determine the amount of iron present in water by spectrophotometric measurements. The method applies Beer-Lambert's Law. A calibration curve is constructed using known concentrations of iron from which unknown concentration of iron can be determined.

Apparatus:

Colorimeter, Volumetric flasks (50 ml.), Burette, Cuvette, filter paper etc.

Chemicals:

Ammonium thiocyanate, ferrous ammonium sulphate, conc. HNO_3, conc. H_2SO_4 etc.

Theory:

Colorimetry deals with measurement of color intensity. The color of a substance is due to absorbance of light waves of certain wavelengths. If solution does not absorb light, it is transparent and colorless. If it absorbs light, it is colored. The absorption of light by solution results in excitation of electrons in its molecule. The advantage of colorimetric analysis is that it requires much less time and it is more accurate than chemical analysis. Colorimetry is used to determine only low concentration usually less than 2%.

Fundamental laws of colorimetry

When light (monochromatic or heterochromatic) is incident upon a homogeneous medium a part of radiant power of incident light (Po)is reflected (Pr) a part is absorbed by medium (Pa) and remainder transmitted (P).

$$Po = Pr + Pa + P$$

Po – Radiant power of incident light.

Pr– Radiant power of reflected light.

Pa– Radiant power of absorbed light.

P– Radiant power of transmitted light.

If a comparison cell or the same cell is used during analysis the value of Pr, which is very small ≈ 4 % can be eliminated for air glass interfaces Po = Pa + P. Colorimetry is based on two fundamental laws, viz; Lambert's law, Beer's law. Combined form of these laws is called as Beer-Lambert's law.

1) Beer's Law

When a beam of monochromatic light is allowed to pass through a transparent medium the rate of decrease of radiant power with the concentration of medium is directly proportional to radiant power i.e. absorbance of solution is directly proportional to concentration of solution.

2) Lambert's Law

This law can be stated as when a beam of monochromatic light is allowed to pass through a transparent medium the rate of decrease of radiant power with thickness of the medium is directly proportional to thickness of medium or path length

3) Beer-Lambert's Law

The combined law states that, Absorbance = constant x thickness of medium x concentration of medium i.e. A= constant x b x c. Thus, laws states that,

When a beam of monochromatic light is allowed to pass through a transparent medium, the absorption of medium is directly proportional to thickness (b) and concentration of medium (c) When the concentration is expressed in mol/lit and thickness in cm,the constant in above equation is called as absorptivity / Molar Extinction Coefficient and represented by symbol'ε'. Thus A = εbc. When b = 1 cm and c = 1 mol/lit then A = ε

The extent to which a sample absorbs light depends strongly upon the wavelength of light. For this reason, spectrophotometry is performed using monochromatic light. Monochromatic light is light in which all photons have the same wavelength. The absorbance spectrum shows how the absorbance of light depends upon the wavelength of the light. The spectrum itself is a plot of absorbance vs. wavelength and is characterized by the wavelength (λ_{max}) at which the absorbance is the greatest. Wavelength is characteristic of each compound and provides information on the electronic structure of the analyte. In order to obtain the highest sensitivity and to minimize deviations from Beers Law, measurements are made using light with a wavelength of λ_{max}. Iron can be estimated by thiocyanate method using Beer Lambert's law.

Thiocyanate Method

Iron (III) reacts with thiocyanate to give a series of intensely red coloured compounds, which remain in true solution, while Iron II does not react.

$$Fe^{3+} + (SCN)^{-}_{n} \longrightarrow [Fe(SCN)_n]^{3-n},\ n = 1 - 6$$

Depending upon thiocyanate concentration, series of complexes can be obtained. In actual practice, large amount of thiocyanate should be used, since this increases the intensity and also the stability of the colour. In presence of strong acids like HCl and HNO_3 (0.05M to 0.5M), hydrolysis of Fe^{3+} ions is suppressed. But SO_4^{2-} ion forms a complex with Fe^{3+}, hence sulphuric acid is not recommended.

Procedure:

Calibration: Set the filter for $\lambda_{max.}$ Prepare the blank solution as under

Blank Solution: Take 5ml solution of NH_4SCN in 50 ml standard volumetric flask. Add 3 ml 2N HNO_3 to it. Dilute the solution up to mark. Shake it well. Fill the cuvette using this solution & adjust the instrument for100 % T or 0% A.

A. Preparation of the solutions for the standard graph/calibration curve

Using common burette add 5, 10, 15, 20 and 25 ml of standard iron solution in different 50 ml volumetric flasks. Add 5 ml solution of NH_4SCN & 3 ml solution of 2N HNO_3 to it. Add water up to the mark. Shake it well. After rinsing the cuvette, fill it with the solution prepared. Measure the absorbance of the solution. Record the absorbance for remaining solutions also. Prepare the observation table II plot a graph of absorbance vs concentration.

B. Determination of concentration of unknown solution

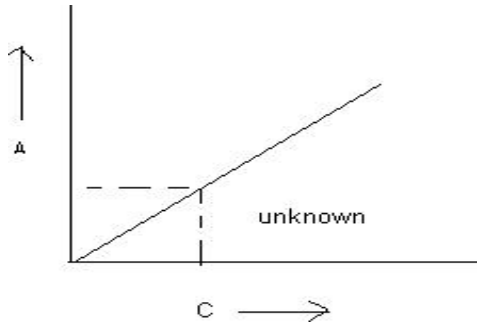
To the given unknown solution, add 5 ml solution of NH_4SCN & 3 ml 2N HNO_3. Dilute up to the mark with water. Shake well. Measure the absorbance of this solution. Plot a graph of concentration (C) vs absorbance (A) – Graph. Find concentration of unknown iron sample from graph.

Precautions:

1. Handle the cuvette carefully.
2. Dry the cuvette from outer side before measuring absorbance of each sample.
3. Calibration of spectrophotometer is needed initially.
4. Do not overfill the cuvette.

Observation table:

Sr. No.	Volume of standard Fe solution in ml	Concentration in mg/ml (C)	Absorbance (A)
1	5		
2	10		
3	15		
4	20		
5	25		
6	Unknown		



Graph of Absorbance (A) Vs. Concentration (C)

Calculations:

Concentration of standard Fe solution = 0.01 mg/ml

Using $C_1V_1 = C_2V_2$, calculate concentration of all solutions. Plot a graph of Absorbance Vs. Concentration

Results:

Concentration of iron present in a given unknown sample = …….mg/ml

Conclusion:

Questions:

1. What is Beer's and Lambert's law?
2. What is the significance of determination of Iron concentration in water?
3. Explain the terms-Absorbance and %Transmittance
4. Explain the basic principle behind Colorimeter.
5. Iron is present in water in which forms? What is their source in drinking water?

Note: Students are instructed to do all necessary calculations and answer the questions on separate sheets and attach them.

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F.Y. B. Tech. Academic Year 2020-21 Trimester:

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Name: _____ **Class:** _____ **Batch:** _____

Roll No.: _____ **Experiment No: 08**

Performed on: _____ **Submitted on:** _____ **Teacher's Sign.:** _____

Aim: Study of removal of heavy metals by adsorbents using UV-visible spectrophotometer

Objective

To study the removal of toxic metals such as copper using certain adsorbents and ascertain the reduction in metal concentration in the solution treated by the adsorbent using UV-visible spectrophotometer.

Apparatus:

UV-Visible spectrophotometer, cuvettes, conical flask, filter paper, funnel etc.

Chemicals:

Copper sulphate hydrate ($CuSO_4.5H_2O$), ammonia solution, activated charcoal, saw dust, distilled water.

Theory:

Excessive release of heavy metals into the environment due to industrialization and urbanization has posed a great problem worldwide. The adsorption process is widely and urbanization the removal of heavy metals from wastewater because of its low cost, availability and eco-friendly nature. Both commercial adsorbents and bio-adsorbents are used for the removal of heavy metals from wastewater, with high removal capacity. In the present study, adsorbents such as activated charcoal and sawdust were employed to adsorb copper ions from aqueous solutions. The solutions were then analyzed for their absorbance on a UV-visible spectrophotometer in order to find out the change in concentration of the copper ions after adsorption. UV-Visible spectroscopy is routinely used in analytical chemistry for the quantitative determination of different analytes, such as transition metal ions, highly conjugated organic compounds, and biological macromolecules. Spectroscopic analysis is commonly carried out in solutions but solids and gases may also be studied. UV-Visible spectroscopy can be used to determine the concentration of the absorbing species in a solution. It is necessary to know how the absorbance changes with concentration.

Procedure

100 ml copper sulphate solution of 50 ppm concentration was prepared in distilled water. 25 ml of the solution was taken in two beakers and 1g each of activated charcoal and saw dust were added to the beakers. The solutions were stirred for 5 minutes and the beakers were left undisturbed for 1 hour at room temperature. After one hour, the solutions were carefully filtered off using Whatman paper. The change in the concentration of copper ions in the solutions, after adsorption, was then analyzed by

using UV-visible spectrophotometer. For this purpose, cupric ammonia complex was prepared by mixing 4 ml of the copper ion solution with 1 ml of ammonia solution which gave a dark blue colored complex. Copper ammine complex adsorbs light around 600 nm. The absorbance of the three solutions (untreated and treated with activated charcoal and saw dust) were plotted against a range of wavelength from 400 nm to 750 nm.

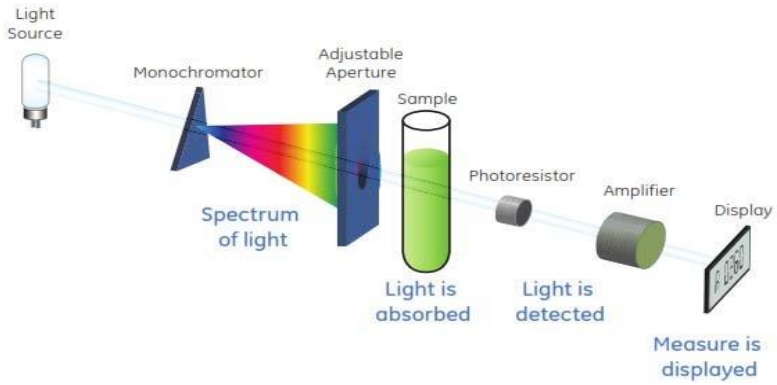

Figure: Schematic diagram of Spectrophotometer

Observation table:

S. No.	Copper sulphate solution*	Absorbance
1.	Untreated	
2.	Treated with sawdust	
3.	Treated with activated charcoal	

* Absorbance of $[Cu(NH_3)]^{2+}$ ions at 600 nm.

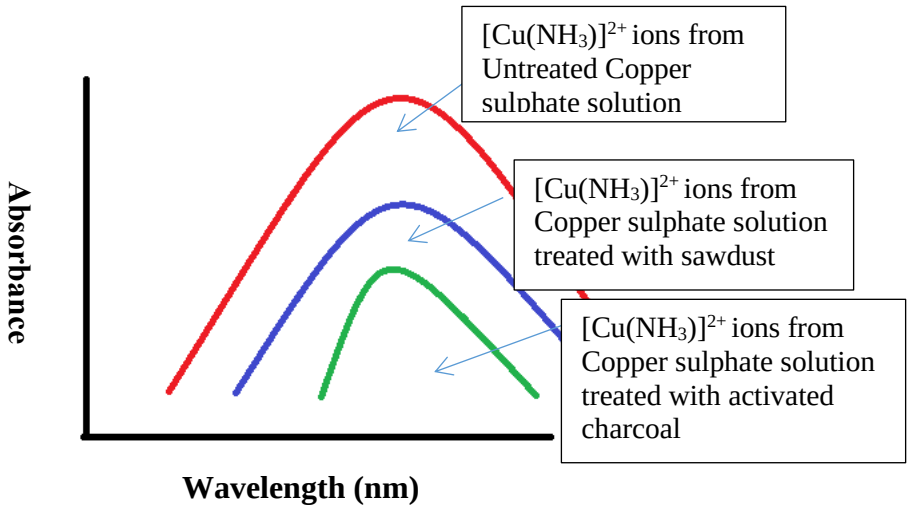

Figure: Plot of Absorbance Vs Wavelength (nm) for three copper sulphate solutions of different concentration.

Result

The present experiment shows that the adsorbents are effective for the removal of copper ions from aqueous solutions. Maximum lowering in concentration of copper ions was observed in the copper solution treated with activated charcoal as shown by the decrease in the absorbance of the solution. Activated charcoal exhibited high adsorption capacity as compared to saw dust.

Conclusion:

Questions

1. Discuss the instrumentation of UV-visible spectrophotometer?
2. Why should we use only distilled water for the experiment?
3. Give examples of some other adsorbents used in removing heavy metals?
4. How does copper salt interact with the adsorbent?

Note: Students are instructed to do all necessary calculations and answer the questions on separate sheets and attach them.

Dr. Vishwanath Karad MIT World Peace University

F.Y. B. Tech. Academic Year 2020-21 Trimester:

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Total =						

Teacher's Signature with Date: **Student's Signature with Date:**

Name: _____. Class: _____. Batch: _____.

Roll No.: _____ Experiment No: 09

Performed on: _____ Submitted on: ____. Teacher's Sign.: _____.

Aim: To determine the molecular weight of a polymer by using Ostwald's Viscometer.

Objective:

To determine the molecular weight of polymer by using Ostwald Viscometer.

Apparatus:

Viscometer, stopwatch, rubber bulb, pipette etc.

Chemicals:

Distilled water, Polyvinyl Alcohol (PVA), Acetone etc.

Theory:

Molecular weight of polymer is always reported as average molecular weight. This can be calculated by measuring the intrinsic viscosity of dilute polymer solution. The molecular weight of the polymer is measured by using viscometer and the molecular weight obtained by this technique is called viscosity average molecular weight. The molecular weight of the polymer solution is very high so the viscosity of polymer solution is very high compared to that of pure solvent.

From the Mark-Houwink equation the relationship among the molecular weight and viscosity are given below

$$[\eta] = KM^{\alpha}$$

Where η is the intrinsic viscosity, M is Molecular weight, K and α αre constants for a particular polymer solvent system.

If we know the K and α values for a given polymer solution the intrinsic viscosity and molecular weight can be calculate using the above equation.

Sr. No.	Polymer	Solvent	K	α
1	Cellulose acetate	Acetone	1.49×10^{-4}	0.82
2	Polystyrene	Toluene	3.7×10^{-4}	0.62
3	Polymethyl methacrylate	Benzene	0.94×10^{-4}	0.76
4	Polyvinyl alcohol	Water	2.0×10^{-4}	0.76

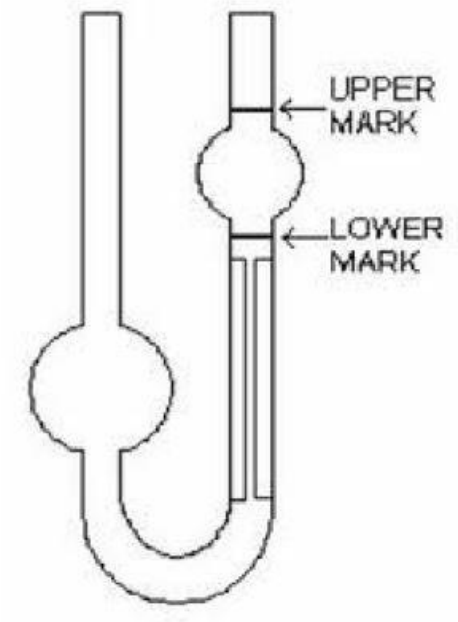


Figure: Ostwald's Viscometer

Procedure:

Take a clean dry viscometer and clamp it in a perfectly vertical position to an iron stand. Clamp the wide arm using paper strip. Attach a rubber tube to the narrow end of the viscometer.

Prepare 1% solution of the given macromolecule using pure solvent. From that prepare 50 ml of 0.5%, 0.4%, 0.3%, 0.2% and 0.1% solutions using standard 50 ml volumetric flask.

Take 25 ml of 0.5%, solution by a pipette and introduce it into the viscometer through the wide arm. Suck the liquid by the rubber tube attached to narrow arm, so that it will rise in the capillary above the mark 'A'. At this stage there should be sufficient liquid left in the bulb of the wider arm. Press the rubber tube firmly by folding it so that the level of the liquid does not get disturbed. Now allow the liquid to flow down through the capillary by releasing the fold on the rubber tube. Note the time required for the flow between the marks A and B. Repeat the procedure three times and find the constant time in seconds.

Clean the viscometer with acetone, dry it and find the time of flow for different dilutions. Finally clean the viscometer and find time of flow for the pure solvent. (Solvent used for the preparation of the solution and dilution)

Observations:

Obs. No.	Solvent / Polymer Solution	Time of 't' (Sec)				$\eta r = \frac{t}{to}$	$\eta_{sp} = \eta_r - 1$	$\eta red = \frac{\eta sp}{C}$	$\eta inh = \frac{(ln \eta r)}{C}$
		I	II	III	Mean				
1	Pure Solvent				t_0				
2	0.5 % Polymer Solution								
3	0.4 % Polymer Solution								
4	0.3 % Polymer Solution								
5	0.2 % Polymer Solution								
6	0.1 % Polymer Solution								

Where as

η_r = Relative viscosity

η_{sp} = Specific viscosity

η_{red} = Reduced Viscosity

η_{inh} = Inherent Viscosity

Calculations:

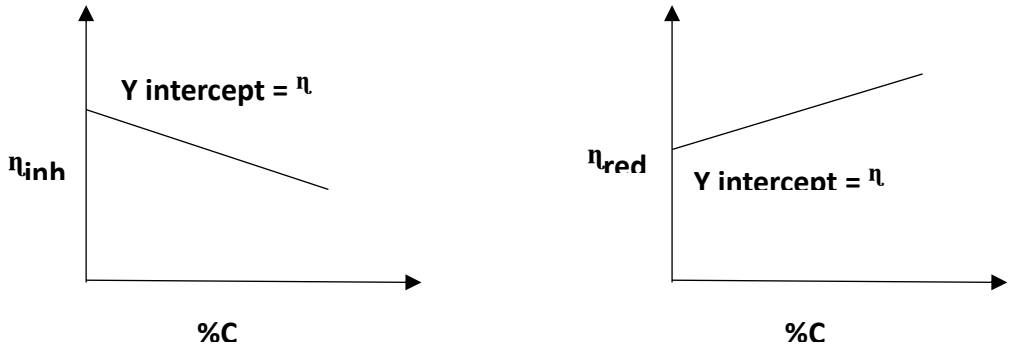
$$\eta_{inh} = KM\alpha$$

$$\log \eta_{inh} = \log K + \alpha \log M$$

$$\log M = \log \eta_{inh} - \log/\alpha$$

$$M = \text{anti}\ (\log\eta - \log K/\alpha)$$

Graphs:



Plot the graph of η_{inh} Vs C and η_{red} vs C

Both the graph gives η value by extrapolating to zero concentration

Result:

1. **Average Molecular weight of polymer by calculation =…….**
2. **Average Molecular Weight by graph =……..**

Conclusion:

Questions:

1. What do you understand by Viscosity?

2. Why PVA is soluble in water?

3. Explain the significance of molecular weight of polymer with respect various properties.

Note: Students are instructed to do all necessary calculations and answer the questions on separate sheets and attach them.

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Teacher's Signature with Date: **Student's Signature with Date:**

Name: _____ **Class:** _____ **Batch:** _____

Roll No.: _____ **Experiment No: 10**

Performed on: _____ **Submitted on:** _____ **Teacher's Sign:** _____

Aim: - Estimation of dissolved oxygen in the given samples of water by Winkler's method.

Objective: To find the amount of dissolved oxygen in the given samples of water by Winkler's method

Apparatus: BOD bottles, Burette, pipette, conical flask, Stand etc.

Chemicals: Water sample, $Na_2S_2O_3$ solution, KI solution, dil H_2SO_4, 0.025N $K_2Cr_2O_7$ solution, distilled water, starch indictor etc.

Theory:

Atmospheric oxygen is not readily soluble in water. Its solubility is directly proportional to its partial pressure. Dissolved oxygen (DO) saturation decreases with rise in temperature, salt concentration, altitude and organic concentration. Decrease of DO with increase in organic contaminants indicate pollution level of water bodies. DO determinations at various points along a river are carried out to define the pollution status of the river. DO level of more than 3 mg/ml is desirable for the existence and growth of fish and such other form of aquatic life. For normal flowing river, DO is in the range of 6 to 8 mg/ml. DO measurements are important for maintaining aerobic conditions in aerobic biological treatment units. DO values are used for corrosion control. For estimation of DO content in a sample, an iodide added to the sample is oxidized under acidic conditions to free iodine. The amount of free iodine liberated is equivalent to the amount of DO originally present in the sample. The liberated iodine is estimated by titrating against standardized sodium-thiosulphate ($Na_2S_2O_3$) solution using starch as an indicator. The amount of free iodine estimated is a measure of DO in the sample.

Procedure:

Part – A: - Standardization of $Na_2S_2O_3$ Solution

Take about 2 g of KI in conical flask. Add 100 ml of distilled water (to suppress sublimation of iodine). Add 10 ml of dil. H_2SO_4 (prepared by taking 9 ml of distilled water and adding 1 ml of conc. H_2SO_4 to it). Add 10 ml of 0.025 N $K_2Cr_2O_7$ solution. Add 100 ml distilled water (to prevent masking of starch end point by greenish trivalent chromium ions).

This is a slow reaction. Wait for 5 minutes to allow all the dichromate added to react completely to release an equivalent amount of free iodine. Titrate it against the $Na_2S_2O_3$ (approx. 0.025N) solution till color changes from blue to colorless. Let the reading be equal to V_1 ml. calculate exact normality N_1 of $Na_2S_2O_3$. Use this reading for the determination of DO.

Observation Table:

Burette : (Approx... 0.025N) $Na_2S_2O_3$ solution

Pipette : 0.025N $K_2Cr_2O_7$ solution

Indicator: Starch solution

End Point: Blue to colorless

Reaction

$$6KI + 7H_2SO_4 + K_2Cr_2O_7 \longrightarrow Cr_2(SO_4)_3 + 3I_2\uparrow + 4K_2SO_4 + 7H_2O$$

Calculations

$Na_2S_2O_3$ $K_2Cr_2O_7$

$N_1V_1 = N_2V_2$

$$\text{Exact Normality of } Na_2S_2O_3 = \frac{N2 \times V2}{V1} = \frac{0.025 \times 10}{V1} = \cdots\ldots N$$

Part B: – DO Fixation

Fill 300 ml BOD bottle completely with the sample. Tap bottle all round to release entrapped air bubbles. Record the temperature. Stopper the bottle. Remove the stopper and add 2 ml of $MnSO_4$, using a pipette-dipping the open end of the pipette below the liquid surface. Add 2 ml of alkali-iodide-azide solution using a pipette dipping the end of the pipette below the liquid level. If DO is absent, a stable white precipitate of manganese hydroxide is formed as per equation.

$$MnSO_4 + 2\ KOH \longrightarrow Mn(OH)_2\downarrow + K_2SO_4$$

The experiment may be stopped at this stage.

If DO is present, manganese ions are oxidized to manganic ions a brown precipitate of manganic basic oxide is formed.

$$Mn(OH)_2 + 1/2\ O_2 \longrightarrow MnO(OH)_2\downarrow$$

Part - C: - Determination of DO

Stopper the bottle containing brown precipitate of manganic oxide and mix by inverting the bottle several times. Remove the stopper and 2 ml of conc. H_2SO_4 using measuring cylinder. Stopper the bottle and mix by inverting the bottle several times units the brown precipitate completely dissolves to yield a uniformly yellow coloured free iodine solution. Under acidic conditions manganic basic oxide oxidizes iodine to free iodine.

$$MnO(OH)_2 + H_2SO_4 + 2\,KI \longrightarrow MnSO_4 + 2\,H_2O + [O]$$

$$2KI + H_2SO_4 + [O] \longrightarrow K_2SO_4 + H_2O + I_2^0$$

Take 203 ml of above iodine solution in a flask, (the extra 3 ml is the correction for 4 ml reagents added for DO fixation). Titrate against std. $Na_2S_2O_3$ Solution using 1 to 2 ml starch indicator.
End point is blue to colourless (X ml).

$$I_2^0 + 2\,Na_2S_2O_3 \longrightarrow Na_2S_4O_6 + 2NaI$$

Sodium tetrathionate

Observation Table

Burette: Std. 0.025N $Na_2S_2O_3$ solution
Flask: BOD bottle solution
Indicator: Starch solution
End Point: Blue to colourless

Sr. No.	Sample	Temp. of sample ^0C	Burette Reading (X ml)
1	Tap Water		
2	Hot tap water		
3	River or lake or well water		
4	Waste water		

Calculations:

$$DO = \frac{X \times N1 \times 8}{200\ (\text{ml of sample taken})} \times 1000 = \cdots \ldots \ldots \ldots \text{mg/L}$$

Result:

Sr. No	Sample	DO, mg/L	Remarks
1	Tap Water		
2	Hot tap water		
3	River or lake or well water		
4	Waste water		

Conclusion:

Questions:

1. What do you understand by the phrase fixation of dissolved oxygen?
2. What is the effect of oxidizing impurities like NO_2^- and Fe^{3+} (if not removed) on the DO results?
3. What is the effect of reducing impurities like SO^{2-}, S^- and Fe^{2+} (if not removed) affect the DO determination?
4. What is the optimum DO value for drinking water as per standard WHO norms?
5. What is the significance of DO measurement?

Note: Students are instructed to do all necessary calculations and answer the questions on separate sheets and attach them.

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