

# **Walchand College of Engineering, Sangli**

*(Government Aided An Autonomous Institute)*

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**Semester-I**

**Group B ( F.Y. B.Tech Civil, Mechanical,  
Electrical and CSE)**

**DEPARTMENT OF CHEMISTRY**

**Engineering Chemistry Lab**

**5CH-151**

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**EXPERIMENT NO 1**

**AIM:** To estimate hardness of given sample of water by EDTA method.

**APPARATUS:** Burette, Pipette, Measuring Cylinder, Titration Flask, Volumetric Flask etc.

**REAGENTS:** CaCO<sub>3</sub> powder, 0.01M EDTA solution, Buffer Solution, Eriochrome Black T Indicator, Water Samples etc.

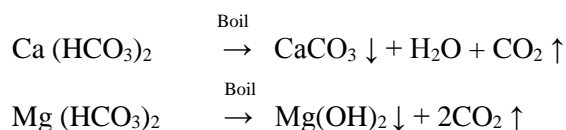
**THEORY: Hardness:-** Caused by dissolved salts of calcium & magnesium, is tendency of water to resist formation of foam or lather with soap solution called as hardness, and kind of water difficultly form foam or lather with soap solution is called hard water. Hardness is expressed in terms of equivalents of CaCO<sub>3</sub>, while it is expressed with units

**1. Part per million (ppm):-** The number part of impure substance present per million (10<sup>6</sup>) parts of water.

**2. Milligrams per liter (mg/l):-** It is number of impure substance in mg present in one liter of water.

$$1\text{ppm} = 1\text{mg/l}$$

**Types of hardness:** - The hardness due to bicarbonate of Ca & Mg is known as **Temporary hardness**. This hardness can be removed by mere boiling.



The hardness due to Chlorides & Sulfates of Ca & Mg is known as **Permanent hardness**. This hardness can't be removed by mere boiling.

Different uses of water demands different specifications. While such type of water cause disadvantages in Textile & Paper industry, Sugar industry, Scale & Sludge formation in boilers, spoiling of dairy food & pharmaceutical products, Increases B.P. of water while cooking food, Consume more soap during bathing & washing applications. Thus hardness estimation is essential to decide suitability of water for domestic and industrial applications.

1-50 Soft, 51-90 Slight hard, 91-190 Moderate hard, 191-250 Hard, 251 -290 Very hard, 291 above Extreme hard water

One of the precise method of estimating total hardness of water is EDTA method (Direct EDTA method from complexometric titration). In this experiment you have to determine hardness of given water samples by using EDTA titration as per the procedure given below.

**PROCEDURE: A) Preparation of Standard Hard Water:** Weigh 0.5 gm. dry CaCO<sub>3</sub> powder and transfer it to 250cc beaker. Dissolve this in minimum quantity of provided nitric acid. Transfer solution to 500cc measuring flask. Rinse beaker with distilled water for at least three times and transfer washing to measuring flask. Dilute the solution to the mark with distilled water and shake well. Transfer 200 cc of this in to a bottle labeled as standard hard water.

**B) Preparation of Standard EDTA solution:** Weigh accurately 1.68g di sodium EDTA. Transfer it to 500cc measuring flask. Add 250cc distilled water and shake well. Dilute with distilled water up to the mark. Transfer 200 cc of this in to a bottle labeled as 0.01M EDTA.

**C) Standardization of EDTA:** Fill the burette with 0.01M EDTA solution. Remove air bubble and adjust zero level. Pipette out 25 cc standard hard water in to titration flask and add to it 2cc buffer solution and 2-4 drops of Eriochrome Black T indicator. Shake well and Titrate with EDTA till Wine red color changes to Sky blue. Take pilot reading & three fine readings.

## OBSERVATIONS

## 5. Pilot Reading

Sr. No	Standard hard water (ml)	Sample Water (ml)
1		
2		
3		
CBR	X=	Y=

= \_\_\_\_\_ ppm hardness of sample.

**Hardness of Water sample = \_\_\_\_\_ ppm**

**EXPERIMENT NO 2**

**AIM:** To estimate extent and type of alkalinity of given sample of water.

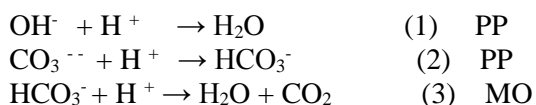
**APPARATUS:** Burette, Pipette, Titration Flask, etc.

**REAGENTS:** N/50 H<sub>2</sub>SO<sub>4</sub>, Methyl Orange, Phenolphthalein Indicator, Water Sample etc.

**THEORY:** Alkalinity of water means the tendency of water to neutralize acids. Natural water present in different sources contain dissolved salts and minerals from soil. Some of these salts cause an increase in concentration of HCO<sub>3</sub><sup>-</sup>, CO<sub>3</sub><sup>2-</sup>, OH<sup>-</sup> ions result alkalinity to water sample. This alkalinity is classified as 1) Caustic or Hydroxyl alkalinity (Due to OH<sup>-</sup>), 2) Carbonate alkalinity (Due to CO<sub>3</sub><sup>2-</sup>) and 3) Bi carbonate alkalinity (Due to HCO<sub>3</sub><sup>-</sup>).

Presence of alkalinity in water cause i) caustic embrittlement in metallic material ii) Formation of Scale & Sludge in boiler tubes & pipes iii) Changes pH of soil iv) Damage constructions on sources of water v) hardness as well as Digestive disorder etc.

The type & extent of alkalinity present in a water sample is determined by using neutralization titration. During this the alkalinity causing ions will neutralize in following sequence.



Reaction 1 and 2 involve titration of strong base and strong acids while reaction 3 involves titration of weak base and strong acid. Thus it needs different Acid-Base indicators. In this experiment the extent and type of alkalinity of given water sample is determined by using following procedure.

**PROCEDURE:** Fill the burette with N/50 H<sub>2</sub>SO<sub>4</sub> solution. Remove air bubble and adjust zero level. Pipette out 25 cc sample water in to titration flask and add to it 2-4 drops of Phenolphthalein indicator. If pink colour is obtained then titrate with N/50 H<sub>2</sub>SO<sub>4</sub> solution till pink color changes to colorless. Record this reading as 'pp'. To the same solution add 2-4 drops of Methyl Orange indicator if yellow colour is obtained then continue titration till yellow color changes to orange red. Record this as reading 'mo'. Take pilot reading and three fine readings for each part. Repeat same procedure for water sample 2.

**OBSERVATIONS**

<b>1. Solution in Burette</b>	--	
<b>2. Solution by pipette</b>	--	
<b>3. Indicator</b>	-- 1.	2.
<b>4. End Point</b>	-- 1.	2.
<b>5. Pilot Reading for</b>	-- pp)	pp)

## OBSERVATION TABLE

Sr. No	Sample Water1 Reading 'pp'	Sample Water1 Reading 'mo'
1		
2		
3		
CBR	ml	ml

## CALCULATIONS –

$$\text{Alkalinity Phenolphthalein (P)} = \frac{\text{Normality of acid} \times \text{Reading pp} \times \text{Eq. wt of CaCO}_3 \times 1000}{\text{Volume of water Sample}}$$

$$P = \frac{1 \times \text{pp} \times 50 \times 1000}{50 \times 25}, \quad \text{i.e. } P = \text{pp} \times 40 \text{ mg / lit}$$

$$P1 = \quad \times 40 = \quad \text{mg / lit}$$

Similarly Alkalinity (Total) (M) = mo\*40 mg / lit.

$$M1 = \quad \times 40 = \quad \text{mg / lit}$$

Determine type of alkalinity and its extent by using following table.

Result of titration to PP end point "pp" & MO end point "mo"	Hydroxide OH <sup>-</sup>	Carbonate CO <sub>3</sub> <sup>--</sup>	Bicarbonate HCO <sub>3</sub> <sup>-</sup>
P= 0	Nil	Nil	M
P=M	M	Nil	Nil
P=1/2 M	Nil	2P	Nil
P> 1/2 M	2P-M	2(M-P)	Nil
P< 1/2 M	Nil	2P	(M-2P)

**Result** – Alkalinity of water sample 1 =                  ppm                  Type –

**EXPERIMENT NO 3**

**AIM:** To estimate Dissolved Oxygen in given sample of water

**APPARATUS:** Burette, Pipette, Titration Flask, Reagent bottle, BOD bottle, beaker, etc.

**REAGENTS:** Water sample, 0.01N  $\text{Na}_2\text{S}_2\text{O}_3$  solution, Conc.  $\text{H}_2\text{SO}_4$ , Starch Indicator, KOH, KI,  $\text{MnSO}_4$  etc.

**THEORY:** DO is an important water quality parameter. The oxygen is sparingly soluble in water. Normally the DO level in water is 10 ppm. If DO is less than 10ppm than water quality is not suitable for drinking purpose & also for aquatic life (animal & plant) will not survive in such water. The lowering of DO is due to:

1. Presence of high traces of ox disable waste in water
2. Lack of contact with air

If DO is very less then water contains high traces of organic matter as oxygen is not available towards its oxidation. Such organic matter is a good food material for growth of harmful bacteria & micro organisms. Therefore drinking water is always estimated for its DO

This DO is estimated by Iodometric titration (Iodine acts as reducing agent). DO from water is taken by an oxygen carrier, and then it is converted in to nascent oxygen. This nascent oxygen liberate equivalent amount of  $\text{I}_2$  by oxidation of KI, which is titrated with standard  $\text{Na}_2\text{S}_2\text{O}_3$ .

Reactions taking place as follows

1.  $\text{MnSO}_4 + 2\text{KOH} \rightarrow \text{Mn(OH)}_2 + \text{K}_2\text{SO}_4$
2.  $2\text{Mn(OH)}_2 + \text{O}_2 \rightarrow 2\text{MnO(OH)}_2$   
Oxygen carrier      Manganic oxide
3.  $\text{MnO(OH)}_2 + \text{H}_2\text{SO}_4 \rightarrow \text{MnSO}_4 + [\text{O}] + 2\text{H}_2\text{O}$   
Nascent Oxygen
4.  $2\text{KI} + [\text{O}] + \text{H}_2\text{SO}_4 \rightarrow \text{K}_2\text{SO}_4 + \text{H}_2\text{O} + \text{I}_2\uparrow$
5.  $2\text{Na}_2\text{S}_2\text{O}_3 + \text{I}_2 \rightarrow 2\text{NaI} + \text{Na}_2\text{S}_4\text{O}_6$

**PROCEDURE:** Wash the DO bottle and dry it. Fill DO bottle with sample water. Add 2 cc saturated solutions of KOH, KI and  $\text{MnSO}_4$  in to sample water by measuring cylinder. Stopper the bottle tightly shake well and keep DO bottle undisturbed for 10-20 minutes till turbidity settle down. After 10-20 minutes shake well and add 1cc of conc.  $\text{H}_2\text{SO}_4$  slowly. Shake well keep steady for 5 minutes. Cool this bottle under running tap water to attain room temperature. Fill the burette with 0.01N  $\text{Na}_2\text{S}_2\text{O}_3$  remove air bubble and adjust zero level. Take 100cc sample with measuring cylinder in to titration flask & immediately stopper DO bottle. Add 3-4 drops of fresh starch indicator and titrate with 0.01 N sodium thiosulphate solution immediately till blue colour disappear. Similarly take another reading. Find mean burette reading.

### OBSERVATIONS:

- |                        |       |
|------------------------|-------|
| 1. Solution in Burette | --    |
| 2. Solution in flask   | --    |
| 3. Indicator           | --    |
| 4. End Point           | --    |
| 5. Burette Reading s   | -- 1) |
|                        | 2)    |

Mean Reading =                      ml (X ml)

### CALCULATIONS:-

1cc of 0.01N  $\text{Na}_2\text{S}_2\text{O}_3 \equiv 0.00008$  gm Oxygen

$\therefore$  'X' cc of 0.01N  $\text{Na}_2\text{S}_2\text{O}_3 \equiv 0.00008 * 'X'$  gm Oxygen ( A )

$\equiv 0.00008 * \quad \equiv \quad$  gm Oxygen

Now 100 cc water sample contain 'A' gm Oxygen

Therefore 1000cc sample  $\equiv 'A' * 10$  gm Oxygen ( B )

$\equiv A * 10$  gm Oxygen =                      gm Oxygen

Now DO in ppm / mg/lit = B \* 1000

= B \* 1000

=                      ppm of Oxygen

**Result:-** Dissolved Oxygen in given water sample =                      ppm.



DATE \_\_\_\_\_

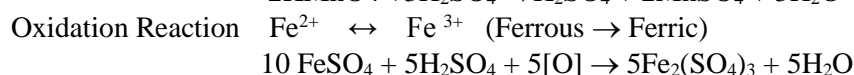
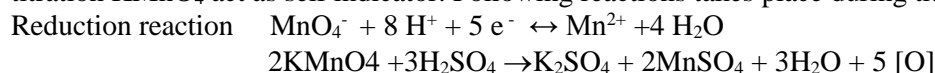
## EXPERIMENT NO 4

**AIM:** To estimate % purity of iron in given sample.

**APPARATUS:** Burette, Pipette, Titration Flask, Reagent bottles etc.

**REAGENTS:** Iron solution, 0.1N  $\text{KMnO}_4$ , dilute  $\text{H}_2\text{SO}_4$  etc.

**THEORY:** This is Redox type of titration. At the beginning of the titration, Iron present in the form of  $\text{Fe}^{2+}$  ion only (100%), while  $\text{Fe}^{3+}$  0%. During the titration, the concentration of  $\text{Fe}^{3+}$  ion increases. At equivalence point, the solution will contain only  $\text{Fe}^{3+}$  &  $\text{Mn}^{2+}$  ions (colourless). Beyond equivalent point, the % of  $\text{MnO}_4^-$  ions (pink colour) will increase therefore color of solution changes from colorless to just pink. In this titration  $\text{KMnO}_4$  act as self indicator. Following reactions takes place during titration.



### PROCEDURE:

- Preparation of standard  $\text{KMnO}_4$  solution – Accurately weigh 1.58g  $\text{KMnO}_4$  and transfer it completely to 500cc measuring flask. Add 200cc distilled water and shake well. Finally dilute it with distilled water up to the mark. Transfer 200cc of this solution to bottle labeled as 0.1N  $\text{KMnO}_4$ .
- Fill the burette with 0.1N  $\text{KMnO}_4$  solution. Remove air bubble and adjust zero level. Pipette out 10 cc iron solution in to titration flask and add to it 20 cc dilute  $\text{H}_2\text{SO}_4$ . Shake well and titrate with 0.1N  $\text{KMnO}_4$  solution (Self indicator) till color changes to just pink. Take pilot reading & three fine readings.

### OBSERVATIONS

1. Solution in Burette	--
2. Solution by pipette	--
3. Indicator	--
4. End Point	--
5. Pilot Reading	--

### OBSERVATION TABLE

Sr. No	Burette readings in ml
1	
2	
3	
CBR	X=

### CALCULATION

1 N 1lit  $\text{KMnO}_4$  solution  $\equiv$  56 gm Iron

Therefore 1 cc 0.1 N  $\text{KMnO}_4$  solution  $\equiv$  0.0056 gm Iron

$$\therefore \text{'X' cc 0.1 N } \text{KMnO}_4 \text{ solution} \equiv \text{'X' * 0.00 56 gm Iron} = \text{'A' gm Iron}$$
$$\equiv \text{'X' * 0.00 56 gm Iron} = \text{_____ gm Iron}$$

Now, 10 cc iron solution contain 'A' gm Iron

100 cc solution contain 'A' \* 10 gm Fe. = 'B' gm Iron

$$= \text{'A' * 10 gm Fe}$$

$$= \text{_____ gm Fe}$$

(Given 'C' = Wt in gm of iron dissolved in 100cc ) Look on black board

$$\therefore \% \text{ of Iron} = \frac{\text{'B' * 100}}{\text{'C'}} \% \text{ Iron}$$

$$= \frac{\text{'B' * 100}}{\text{'C'}}$$

**Result:** % purity of Iron in given sample = \_\_\_\_\_ %.

**EXPERIMENT NO 5**

**AIM:** To determine viscosity of given liquid.

**APPARATUS:** Ostwald's Viscometer, Stop watch, Air dryer etc

**REAGENTS:** Given liquid, Distilled water, Acetone etc.

**THEORY:** It is property of liquid or fluid by virtue of which it offers resistance to its own flow. The flowing liquid consists of a series of parallel layers moving one over other. Any two layers will move with velocity of different magnitudes. Top layer moves faster than the next lower layer, due to internal friction called viscous drag. The unit of viscosity is poise & is defined as, when force of one dyne is required to maintain a relative velocity difference of 1cm/sec between two parallel layers, separated by 1cm the coefficient of viscosity of a liquid is 1 poise. A smaller corresponding unit is centipoise.

**1poise = 100 centipoise.**

**Properties of lubricants**

**1. Viscosity:** It is property of fluid, by virtue of which it resists its own flow.

It is most important single property of lubricating oil. Selection of lubricant is based on their viscosity. If viscosity of lubricating oil is too low, it cannot maintain required lubricating film. & if viscosity of lubricating oil is too high excessive pressure will result.

Therefore select lubricant with adequate viscosity for proper working condition, since lubrication is being done.

**2. Viscosity Index: (V.I.)** It is arbitrary scale to measure variation in viscosity with respect to change in temperature.

As per changes in viscosity of lubricating oil occurs, they are classified into two categories Oil with High VI & Oil with Low VI called as a) High VI lubricants & b) Low VI lubricants

a) High VI lubricants: There is no change in viscosity or very small change in viscosity with very high change in temperature is called High VI lubricants.

b) Low VI lubricants: There is very large change in viscosity with small change in temperature or very high change in temperature is called Low VI lubricants.

Simple method for estimate viscosity of any liquid at room temperature is Ostwald's Viscometer. In this method fixed volume of liquid & pure water allow to flow through a standard capillary tube in a fixed distance. The time in second taken by liquid & pure water is a comparative parameter to find out viscosity of unknown liquid at room temperature

**PROCEDURE:** You are provided Ostwald viscometer. Clean and dry it with acetone. Fill it with distilled water from reservoir end such that level of water must touch the lower end of capillary. Place viscometer on stand provided. Ensure that viscometer is not inclined. Suck water through rubber tube just above upper mark of sample holder bulb present above capillary. Adjust water level to mark 'A'. Keep stop watch ready. Record time required for water level to travel from mark A to mark B. Take three readings. Repeat the same procedure for given liquid.

## OBSERVATION TABLE

Name of Liquid	Sp. Gravity	Time required in sec.	Mean time in sec
Distilled water	1	1 2 3	
Liquid Benzene / Aniline	0.878/ 1.022	1 2 3	

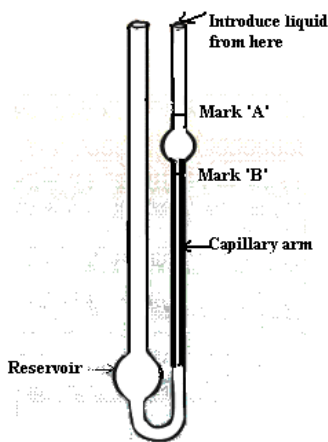
## CALCULATIONS

$$\text{Viscosity of liquid } \eta = \frac{d_o * t_o}{d_w * t_w} \times \eta_w \text{ Centipoise}$$

$$\text{Viscosity of liquid } \eta = \frac{\quad \quad \quad}{\quad \quad \quad} \times 0.89 = \quad \quad \quad \text{Centipoise}$$

**Result:** Viscosity of given liquid =  $\quad \quad \quad$  Centipoises at  $\quad \quad$  °C

Where,  $\eta_w$  = Viscosity of Water (0.89),  $d_o$  = density of benzene /Aniline,  $d_w$  = density of water,  $t_o$  = time for benzene,  $t_w$  = time for water.



**Ostwald Viscometer**

**EXPERIMENT NO 6**

**AIM:** To estimate % of Cu from given bronze solution.

**APPARATUS:** Burette, Pipette, Titration Flask, Reagent bottle with bronze solution, beaker, etc.

**REAGENTS:** Bronze solution, 0.1N  $\text{Na}_2\text{S}_2\text{O}_3$  solution, 5%  $\text{Na}_2\text{CO}_3$  Solution, 5N Acetic acid solution, 10% KI solution, Starch indicator etc.

**THEORY:** Bronze is broad term which defines an alloy of copper with elements other than Zn & Ni. Bronze is basically an alloy of copper & tin. Based on composition of different elements the different properties of alloy will be controlled & bronzes can be divided into different types.

**Phosphor bronze:** 90% Cu, 10% Sn, 0.5% P Used as bearing metal.

**Aluminum bronze:** 88-91% Cu, 0.35% Sn, 6-8% Al, 1-3% Fe 1% Mn Used as bearings, gears, valves etc.

**Silicon bronze:** 90-92% Cu, 1% Sn, 0.5-0.1% Fe, 0.2-1% Mn, 1-4% Si. Used for marine hardware

The estimation of alloy for its composition is useful to predict the mechanical properties like strength, toughness, hardness, malleability, ductility etc. As bronze is containing Cu as parent element the amount of Cu decide most of the properties. Their chemical analysis involves Iodometric titration. Cu ions present in solution react with KI & liberate equivalent amount of  $\text{I}_2$  which is titrated with standard  $\text{Na}_2\text{S}_2\text{O}_3$ .

**Preparation of solution:** First a known weight of an alloy ("p"gm) is dissolved in hot concentrated nitric acid (minimum quantity) & then solution is diluted up to 250cc by distilled water & transfer in reagent bottle.

**PROCEDURE:** Fill the burette with 0.1N  $\text{Na}_2\text{S}_2\text{O}_3$  solution. Remove air bubble and adjust zero level Pipette out 25 cc bronze solution in to titration flask and add to it 5%  $\text{Na}_2\text{CO}_3$  solution drop wise till permanent turbidity  $\text{Cu}(\text{OH})_2$  is formed.. Dissolve this turbidity by adding minimum quantity of 2N acetic acid solution. In above solution add 10cc of 10% KI solution & immediately titrate up to 7cc. Add 1-2 cc fresh starch indicator & continue titration till color changes from blue to colorless. (Supernatant liquid) Take pilot reading and three fine readings by immediately titrating up to initial level of pilot reading.

**OBSERVATIONS:**

1. Solution in Burette      --
2. Solution by pipette      --
3. Indicator      --
4. End Point      --
5. Pilot Reading      --

**OBSERVATION TABLE**

Sr. No	Burette readings in ml
1	
2	
3	
CBR	X=

**CALCULATIONS**

1cc of 0.1N  $\text{Na}_2\text{S}_2\text{O}_3 \equiv 0.006357 \text{ gm Cu}$

$\therefore$  'X' cc of 0.1N  $\text{Na}_2\text{S}_2\text{O}_3 \equiv 0.006357 * 'X' \text{ gm Cu (A)}$

$\equiv 0.006357 * 'X' \text{ gm Cu} \equiv \text{_____ gm Cu}$

Now 25 cc alloy solution contain 'A' gm Cu

Therefore 250 cc alloy solution  $\equiv 'A' * 10 \text{ gm Cu (B)}$

$\equiv 'A' * 10 \text{ gm Cu}$

$= \text{_____ gm Cu}$

$$\% \text{ of Cu} = \frac{100 * B}{P} = \frac{100 * B}{P}$$

Where P = Weight of alloy dissolved in 250 cc solution. (See black board)

% of Cu = \_\_\_\_\_

**Result:** % of Cu in given sample = \_\_\_\_\_ %.

**EXPERIMENT NO 7**

**AIM:** To perform acid base titration pH metrically

**APPARATUS:** Glass electrode, beaker, pH meter etc.

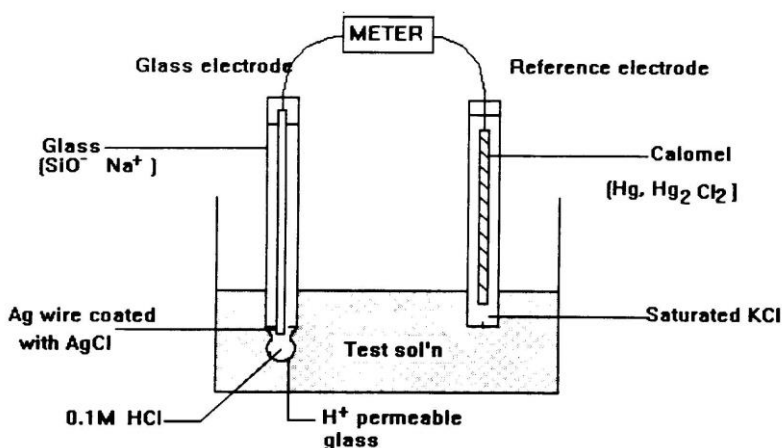
**REAGENTS:** Given acid (Bottle A) and Base (Bottle B)

**THEORY:** The modern pH meter is an electronic digital voltmeter scaled to read pH directly and may range from a comparatively simple handy instrument suitable for use in the field also. It is useful technique to find out

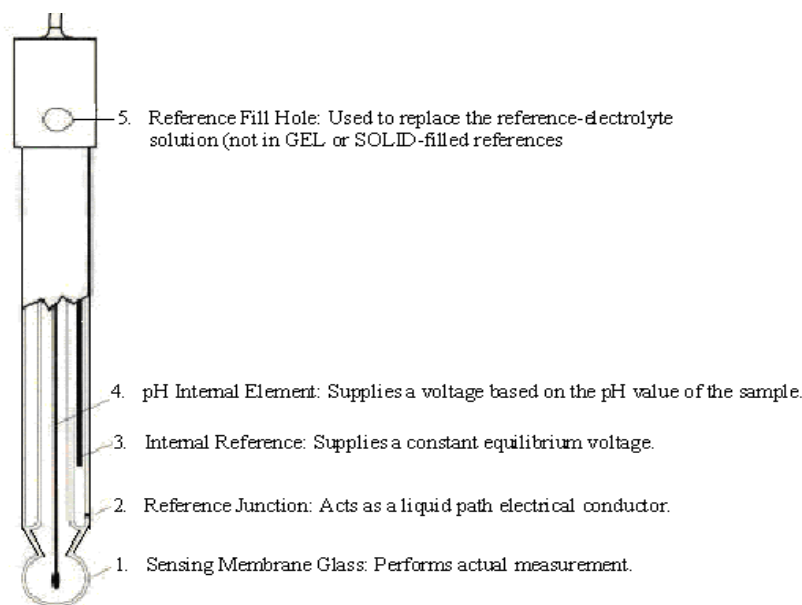
1. Equivalent point of any type of Acid – Base titrations
2. To carry out above titrations in presence of strong oxidant, reductants, viscous protein mediums, as well as colored, colloidal, complex, poisonous solutions.
3. To carry out weak acid weak base titrations or for dilute solutions.
4. To carry out equivalent point of mixtures of two or more acids or bases in single titration.
5. To find out equivalence point of di & tri basic or acidic substances in single titrations.
6. To find out equivalence point of acid base titration in non aqueous medium.

**DIAGRAM:**

**SCHEMATIC DIAGRAM OF pH METER**



## Glass Electrode Diagram.



Glass electrode: Lower bulb of glass electrode is highly sensitive to  $H^+$  ion concentration. It is made up of Corning glass. Thickness of the walls of glass bulb varies from 0.001 – 0.05 mm. It possesses low M.P. & high electrical conductivity. It is filled with 0.1M HCl solution and Ag wire coated with AgCl immerse in it. It is represented as Pt or Ag/ AgCl / 0.1 N HCl / Glass bulb/ Test solution.

When glass electrode immersed in unknown solution, due to variation in concentration of  $H^+$  ions inside & out side of the bulb potential is developed on inner & outer wall of glass bulb due to exchange of  $H^+$  & free  $Na^+$  present in crystal lattice of glass bulb, denoted by  $E_1$  &  $E_2$  potential. Difference between this two potential is called boundry potential. This potential difference is measured with potentiometer. This is displayed as pH on display of digital pH meter.

### Limitations:

1. No use of ordinary potentiometer cost of equipment increases.
2. Glass is fragile, handle with care.
3. Calibration needed frequently.
4. Accuracy depends on reference used for calibration.
5. Not applicable in de hydrating solutions.

### Procedure for pH- metric titration:-

- 1) Clean the electrode by distilled water
- 2) Take 25 ml of given acid solution by pipette in a 50 ml beaker and immerse the electrode. Note down the pH meter reading for zero ml base addition.
- 3) Fill the burette with standard base solution (0.1N NaOH). Remove air bubble and adjust zero level.
- 4) Add Standard base solution gradually from the burette i.e. 0.5 ml at a time to acid solution, stir the same for 1 minute. Note the corresponding value of pH. Record readings in table given.



**OBSERVATION TABLE (B)**

Volume of 0.1N NaOH added in ml	Observed pH	$\Delta v$	$\Delta pH$	$\Delta pH / \Delta v$	$\Delta^2 pH / \Delta v^2$
0		---	---	--	--
0.5					
1					
1.5					
2					
2.5					
3					
3.5					
4					
4.5					
5					
5.5					
6					
6.5					
7					
7.5					
8					
8.5					
9					
9.5					
10					
10.5					
11					
11.5					
12					
12.5					
13					

Find equivalence point by plotting graphs of a) pH vs Volume b)  $\Delta pH / \Delta v$  vs volume and  
c)  $\Delta^2 pH / \Delta v^2$  vs volume added

**CALCULATION:**

Equivalence point from graph. a) = see graph 1 ml b) = see graph 2 ml c) = see graph 3 ml

Normality of Acid =  $N_1V_1 = N_2V_2 = 0.1 \times \text{Equivalence point c} / 25$

=

= N

**Result : 1) Normality of Acid** \_\_\_\_\_ N

**2) pH range at equivalence point** \_\_\_\_\_

**3) Type of acid base titration** \_\_\_\_\_

**EXPERIMENT NO 8**

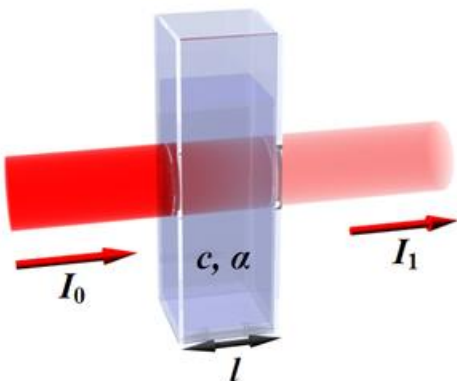
**AIM :** To estimate Copper in given sample Calorimetrically/ Spectrometrically

**APPARATUS :** Colorimeter/ Spectrometer, volumetric flasks etc.

**REAGENTS:** Copper sulphate solution, Ammonia solution.

**THEORY:**

The working of colorimeter/ Spectrometrically is based on Beer's- Lambert's law. It state that, When beam of monochromatic light passing throw homogeneous absorbing medium, decrease in intensity of light is directly proportional to intensity of incident light as well as concentration of absorbing medium.



**Transmission Or Transmittance: (T):** It is simple ratio of intensity of transmitted light to incident light

$$\text{i.e. } T = I_t / I_o$$

**Optical density (OD) or Absorbance (A):** It is simple logarithmic ratio of intensity of incident light to transmitted light. **OD or A = log I<sub>o</sub>/I<sub>t</sub>**

In spectroscopy, the **absorbance A** is defined as

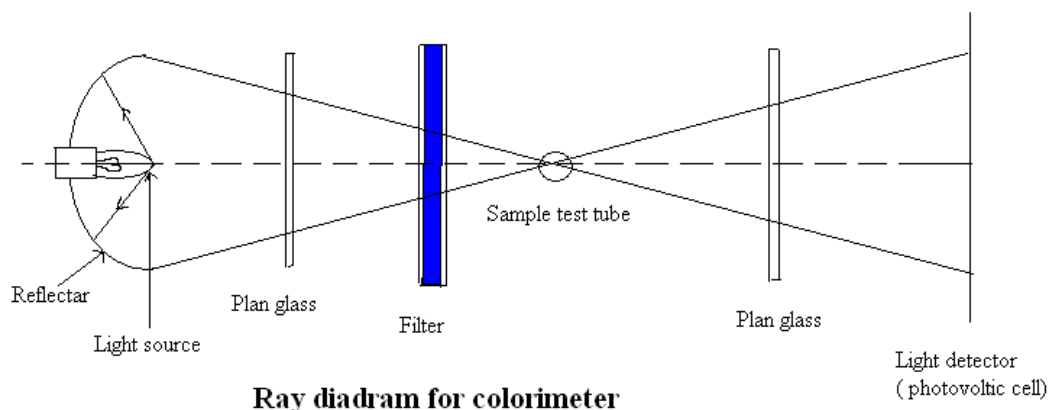
$$A_{\lambda} = \log_{10}(I_0/I),$$

where  $I$  is the intensity of light at a specified wavelength  $\lambda$  that has passed through a sample (transmitted light intensity) and  $I_0$  is the intensity of the light before it enters the sample or incident light intensity.

Absorbance measurements are often carried out in analytical chemistry, since the absorbance of a sample is proportional to the thickness of the sample and the concentration of the absorbing species in the sample, in contrast to the transmittance  $I / I_0$  of a sample, which varies exponentially with thickness and concentration.

See the Beer-Lambert law for a more complete discussion

The schematic ray diagram of colorimeter/ Spectrometrically is as shown below

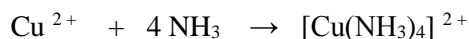


### Applications of Colorimetry

1. Concentration of solution as low as  $10^{-7}$  M which cannot be determined by the conventional method like volumetric & gravimetric.
2. Determination of iron.
3. Determination of ammonia using Nessler's reagent.
4. Determination of aluminum using Erio-chrome cyanine
5. Determination of arsenic by molybdenum blue method.
6. Simultaneous determination of chromium and manganese in steel.
7. Analysis of ore, minerals, alloys & other industrial raw materials & finished products.
8. Analysis of environmental samples.
9. In determination of molecular composition of complexes.
10. For studying Cis & Trans isomers.

### PROCEDURE:

1. You are provided with stock solution of copper.
2. Place the given stock solution in a burette & transfer 05, 10, 15, 20 ml of this solution into separate labeled 100 ml volumetric flasks.
3. Add 5 ml ammonia solution to them & make the volume up to the mark using distilled water.
4. Stopper the flask & shake the solution well.



Copper ion    Ammonia        Deep blue complex

5. Make a blank solution by diluting 5 ml  $\text{NH}_3$  solution in a 50 ml volumetric flask. Add distilled water up to the mark & mix well.
6. Selection of  $\lambda_{\text{max}}$  or filter: a) Select filter no.1, Insert blank solution test tube in a sample holder & Press % T button. Followed by pressing unlock button & then calibrate button. Observe 100 on display.

Immediately remove blank test tube and insert test tube containing 100ppm solution from flask 5. Press **ABS** button and record reading in table 1. Repeat above procedure for all filters, and record readings.

From table no. 1 find out filter at which **ABS** is maximum. Select that filter as  **$\lambda$  max**.

7. Select this filter, calibrate with blank solution and record **ABS** for all solutions.( 2, 3, 4, 5, and unknown) using test tube used for blank solution. Record readings in table 2. .

c) Plot a graph between ABS (OD) & Concentration. Find out unknown concentration graphically.

X axis: Concentration of Cu in mg                      Y axis: OD

Nature of graph: Straight line passing through origin.

### OBSERVATION TABLE

**Table No.1: For  $\lambda$  max**

**Table2**

Sr. No.	Filter (nm)	Optical Density (ABS)	Vol. of std. Sol	Conc. mg/l	OD at $\lambda$ max
1	400		05ml	25	
2	420		10ml	50	
3	470		15ml	75	
4	500		20ml	100	
5	530		Test Solution	Unknown	
6	620		Conc. of Cu in unknown solution from Graph  -----mg/l		
7	660				
8	700				

**Result: Concentration of unknown solution is \_\_\_\_\_ mg/l**

## QUESTION BANK – (FOR QUIZ AT END OF LAB COURSE)

### 1. Draw neat labeled Diagram of

Glass electrode, Ostwald's viscometer, Single beam spectrometer, pH meter setup

### 2. Give reasons

- i) Hardness of water / Water quality parameters are measured as equivalents of  $\text{CaCO}_3$ .
- ii) When  $P=1/2M$  alkalinity is only due to carbonate.
- iii) If  $P=M$  then alkalinity is only due to  $\text{OH}^-$ .
- iv) The sample water should not be exposed to air during DO estimation.
- v) Temporary hardness vanishes on mere boiling.
- vi) Bronzes are known as bearing metals.
- vii) Colorimetric estimation requires monochromatic light.
- viii) Determination of  $\lambda_{\text{max}}$  is a must during colorimetry.
- ix) Strong acid strong base titration can use any acid base indicator.
- x) Viscosity of liquid changes with temperature.
- xi)  $\text{KMnO}_4$  acts as self-indicator during iron estimation.
- xii) Iodometric titrations can be conducted without indicator.
- xiii)  $1\text{ ppm} = 1\text{ mg/L}$
- xiv) Weak acid weak base titrations are normally avoided.
- xv) Hard water gives foam/lather difficultly with soap.
- xvi) Metal indicator complex must be less stable than metal EDTA complex.
- xvii) Hardness estimation by EDTA needs strong basic buffer.
- xviii) Alkalinity due to  $\text{OH}^-$ ,  $\text{CO}_3^{2-}$  and  $\text{HCO}_3^-$  will never exist with any water sample.
- xix) pH range at end point for acid base titration must be known.
- xx) Std. solution prepared with primary standard substance is desirable in titrimetry.
- xxi) Slight excess precipitant is required during precipitation.

### 3. Solve the following

- i.) What is the percentage by mass of a solution made by dissolving 0.49 g of potassium sulfate in 12.70 g of water?
- ii.) What is the percentage concentration of 75.0 g of ethanol dissolved in 500.0 g of water?
- iii.) A chemist dissolves 3.50 g of potassium iodate and 6.23 g of potassium hydroxide in 805.05 g of water. What is the percentage concentration of each solute in the solution?
- iv.) A student wants to make a 5.00% solution of rubidium chloride using 0.377 g of the substance. What mass of water will be needed to make the solution?
- v.) Determine the molarity of a solution prepared by dissolving 141.6 g of citric acid,  $\text{C}_3\text{H}_5\text{O}(\text{COOH})_3$ , in water and then diluting the resulting solution to 3500.0 mL.

- vi.) What is the molarity of a salt solution made by dissolving 280.0 mg of NaCl in 2.00 mL of water? Assume the final volume is the same as the volume of the water.
- vii.) What is the molarity of a solution that contains 390.0 g of acetic acid,  $\text{CH}_3\text{COOH}$ , dissolved in enough acetone to make 1000.0 mL of solution?
- viii.) What mass of glucose,  $\text{C}_6\text{H}_{12}\text{O}_6$ , would be required to prepare  $5.000 \times 10^3$  L of a 0.215 M solution?
- ix.) What mass of magnesium bromide would be required to prepare 720. mL of a 0.0939 M aqueous solution?
- x.) What mass of ammonium chloride is dissolved in 300. mL of a 0.875 M solution?
- xi.) How many moles of  $\text{H}_2\text{SO}_4$  are in 2.50 L of a 4.25 M and 4.25 N aqueous solution?
- xii.) Determine the molal concentration of 71.5 g of linoleic acid,  $\text{C}_{18}\text{H}_{32}\text{O}_2$ , in 525 g of hexane,  $\text{C}_6\text{H}_{14}$ .
- xiii.) You have a solution that is 16.2% sodium thiosulfate,  $\text{Na}_2\text{S}_2\text{O}_3$ , by mass.
  - a. What mass of sodium thiosulfate is in 80.0 g of solution?
  - b. How many moles of sodium thiosulfate are in 80.0 g of solution?
  - c. If 80.0 g of the sodium thiosulfate solution is diluted to 250.0 mL with water, what is the molarity of the resulting solution?
- xiv.) What mass of anhydrous cobalt(II) chloride would be needed in order to make 650.00 mL of a 4.00 M cobalt(II) chloride solution?
- xv.) A student wants to make a 0.150 M aqueous solution of silver nitrate,  $\text{AgNO}_3$  and has a bottle containing 11.27 g of silver nitrate. What should be the final volume of the solution?
- xvi.) What mass of urea,  $\text{NH}_2\text{CONH}_2$ , must be dissolved in 2250 g of water in order to prepare a 1.50 *m* solution?
- xvii.) What mass of barium nitrate is dissolved in 21.29 mL of a 3.38 M solution?
- xviii.) Describe what you would do to prepare 100.0 g of a 3.5% solution of ammonium sulfate in water.
- xix.) What mass of anhydrous calcium chloride should be dissolved in 590.0 g of water in order to produce a 0.82 *m* solution?
- xx.) How many moles of ammonia are in 0.250 L of a 5.00 M aqueous ammonia solution? If this solution were diluted to 1.000 L, what would be the molarity of the resulting solution?
- xxi) Nickel in Nichrome is determined by gravimetry. 0.550g of an alloy is dissolved in acid & diluted by distilled water to get 100cc solution. Nickel from solution is ppted as  $\text{Ni}(\text{C}_4\text{H}_7\text{O}_2\text{N}_2)_2$  weighted 0.660g. Calculate %W/W of Ni. ( Ni-58.7     $\text{Ni}(\text{C}_4\text{H}_7\text{O}_2\text{N}_2)_2 - 288.03$ )

#### 4. Answer the following

- a. Discuss requirements of complexing agent for complexometry

- b. Discuss EDTA as versatile complexing agent for complexometry.
- c. Give an account of different types of EDTA titrations
- d. What is metal ion indicator? Explain its working.
- e. Give requirements of metal ion indicator.
- f. Define chemical analysis and give an account of its classification.
- g. What are standards in titrimetry? Discuss requirements of primary standards.
- h. Discuss requirements of chemical reaction in titrimetric analysis.
- i. What is titrimetry? Give an account of classification of titrimetric methods
- j. Explain working of an acid base indicator.
- k. How a proper acid base indicator is chosen for neutralization titration.
- l. Discuss requirements for acid base indicator.
- m. What are iodine titrations? Discuss in brief iodometry.
- n. Give properties, composition and uses of pig iron, cast iron and wrought iron.
- o. Define an alloy. What are purposes of alloying?
- p. What are bronzes? Give composition, properties and uses of phosphor bronze, gun metal, aluminum bronze and silicon bronze.
- q. Discuss significance of dissolved oxygen
- r. Draw structure of EDTA and EDTA metal complex
- s. Discuss role of viscosity and viscosity index in selection of lubricant
- t. State applications of viscosity measurement
- u. State Beer's Lambert law and give applications of colorimetry
- v. Distinguish between primary and secondary standards.
- w. Give significance of estimating purity of metal with example
- x. Explain why alloys are estimated for its chemical composition.
- y. Write chemical reactions taking place during DO estimation.
- z. State composition, properties and uses of different examples of Brass & Bronzes
- aa. Classify type's volumetric analysis.

## 5. Define following terms

Chemical analysis, Qualitative analysis, Quantitative analysis, Volumetric/Titrimetric analysis, Standard, Primary standard, Titrant, Titrand, Titration, Indicator, Equivalence point, Stoichiometric end point, Theoretical end point, Practical end point, Solution, Solvent, Solute, Dilute solution, Concentrated Solution, Saturated solution, Concentration of solution, Normality, Gram equivalent, Molarity, Mole, Molality, Acidimetry, Alkalimetry, Acid base indicator, Complex, Complexing agent, Metal ion indicator. Acid, Strong acid, Hardness,



Hard water, Temporary hardness, Permanent hard water, Alkalinity of water, acidity of water,  $\lambda_{\text{max}}$ , monochromatic light, absorbance, transmittance, pH, pOH, Viscosity, viscosity index, buffer.

**Note -**

**These are some of sample questions for quiz. Other questions of above types are possible.**