Department of Civil Engineering

Details of Lesson Plan

S.No.	Particulars	Details
1.	Course Name	Engineering Chemistry Lab
2.	Course Code	BSC-113
3.	Academic Year	2024-25
4.	Semester	1st
5.	Number of Lesson plans	10
6.	Faculty Assigned	Dr. KavitaAbrol

Faculty Signature

EXPERIMENT -1

AIM:Find the total hardness of a sample of water by Complexometric method (.EDTA) :

APPARATUS:

Burette,Pipette,250 ml beakers,Conical flasks,Burette stand.

REQUIREMENT:

0.5 M EDTA solution,Indicator Eriochrome Black-T,Water sample and buffer solution (NH4Cl-NH4OH).

THEORY:

The hardness by complexometric method involves use of EDTA as a complexing agent. The Ca ++ and Mg++ present in water are titrated with EDATA using EBT (Ind) at pH10(Buffer).

i) Ca++/ Mg++ $\longrightarrow$ [Ca++/Mg++ - EBT]
Unstable complex, wine red Color)

ii) ii) [Ca++/Mg++ - EBT] + EDTA $\longrightarrow$ [Ca++/Mg++ - EDTA] + EBT
Stable complex Blue Color

Appearance of Blue color indicates the complexation of metal ions with EDTA and the volume of EDTA is used for calculation of hardness of water.

PROCEDURE: Rinse and fill the burette with EDTA solution and note the initial reading .Pipette out 10 ml of standard hard water in the titration flask, To it add 2ml of buffer solution and 3 drops of Eriochrome black T indicator. The color of the solution turns wine red. To this solution add EDTA solution drop by drop with constant shaking till the color changes to blue. Note the final reading. This will give the volume of the EDTA used. Repeat the process twice to get three concordant readings .Let the volume of EDTA used is V1 ml

Now pipette out 10 ml of unknown water sample and titrate it with EDTA as done above using buffer and indicator and record three concordant readings ,Let the volume of EDTA used is V2 ml.

OBSERVATIONS

Observation Table –I (For Standard Hard water)

S.No	Burette Readings		Volume of EDTA used
	Initial Reading	Final reading	(Final-Initial Reading)
1			

2			
3			

Concordant Reading=V1 ml

Observation Table –II (For Unknown water sample)

S.No	Burette Readings		Volume of EDTA used (Final-Initial Reading)
	Initial Reading	Final reading	
1			
2			
3			

Concordant reading V2= ----- ml

CALCULATION:

1 ml of Standard hard water contains CaCO3= 1mg

V1 ml of EDTA solution is consumed by 10 ml of Standard Hard water= 10 mg CaCO3

1 ml of EDTA solution = 10/V1 mg CaCO3

V2ml of EDTA is consumed by 10 ml Unknown water sample

$$\frac{10xV2}{V2ml\ EDTA = V2}\ \text{gm CaCO3}$$

Now 10 ml of unknown water sample contain CaCO3== $\frac{10xV2}{V2}$ ---------- gm CaCO3

1000ml of unknown water sample contain CaCO3 = $\frac{10xV2x1000}{V1\ x10}$

Total Hardness of water sample = -----mg/L or ppm

EXPERIMENT -2

AIM: To Determine the method of purification of organic compounds by Column chromatography .

APPARATUS:

A vertical column,conical flask, funnel, glass rod and measuring cylinder.

CHEMICALS REQUIRED: dry silica gel, organic compounds, suitable solvent like ethyl acetate and petroleum ether and cotton

THEORY:

The organic compounds when prepared commercially have little bit of impurities in their compositions. These impurities from these compounds can be separated by column chromatography. In this technique different impurities as well as pure compounds adsorbs on the surface of the silica gel with different adsorbing forces .Then these adsorbing substances are run through different solvents (usually polar) and the impurities which are adsorbed with less force come down first and then the pure organic compound.

PROCEDURE:

The given organic compound is mixed thoroughly with the dried silica gel in presence of inert solvent. The solvent is dried on the water bath and slurry (comp + silica) is made. This slurry is then packed in column having cotton already packed at the bottom with the solvent. When the slurry is packed , a piece of cotton is placed above so that the solvent may pass smoothly when solvent is passed the impurities start moving downwards with the solvent which is collected in the conical flask. The finishing of the impurities and the separation of the pure product is determined by Thin Layer Chromatography (TLC).The pure compound is collected in the conical flask with the solvent . The solvent is dried and pure organic compound is crystallized.

PRECAUTIONS:

1) The glass column should be cleaned and pre- dried.
2) It should remain vertical throughout the experiment.
3) The silica gel should be dried i.e. it is pre- heated.
4) The level of solvent should always be higher that the level of slurry in the column.
5) The solvent collection in flask should be done drop by drop.

EXPERIMENT -3

AIM: TO PREPARE PURE AND DRY SAMPLE OF ASPIRIN (ACETYL SALICYLIC ACID)

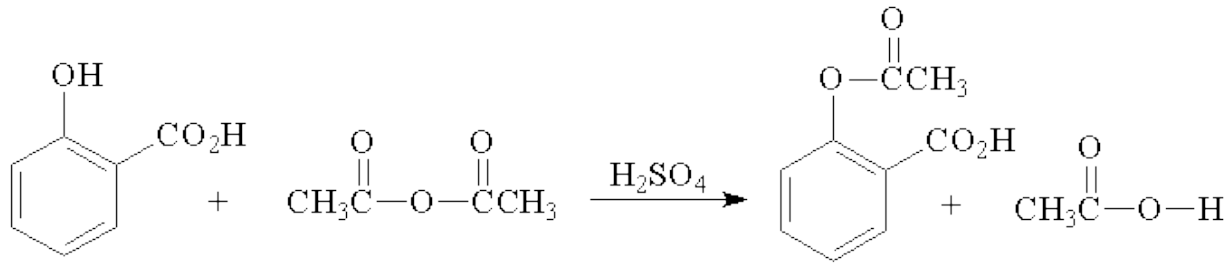
APPARATUS :

Conical flask (100 cm^3).Water bath, Beakers,Glass rod.

CHEMICALS REQUIRED:

Salicylic acid, acetic anhydride, sulphuric acid, acetyl chloride , and alcohol,

REACTION:



PROCEDURE:

1. Take 5 gms of salicylic acid in 100 ml conical flask. To this add about 10 ml of acetic anhydride and 1-2 drops of sulphuric acid.
2. Shake the content thoroughly; the temperature will rise to 70 -80^oC because of the exothermic reaction. Maintain the temperature to 60 – 70^oC for about 15 minutes keeping on water bath.
3. Allow the solution to come down to room temperature to room temperature and then our it in 100 ml of cold water taken in 500 ml beaker with stirring (water is added to destroy excess acetic anhydride which gets converted to acetic acid).
4. To induce crystallization scratch the side of the flask with the help of glass rod.
5. Filter the solid,wash it with cold water. Dry it between the folds of filter paper.
6. TO obtain colorless crystal recrystallization can be done using equal volumes of ethanol and water.

7. Determine the melting point of the crystallized sample.

RESULT:

Yield = ----------

M. pt. = ---------- ^{o}C(130-135 ^{o}C)

PRECAUTIONS:

1) Take acetic anhydride in excess as it act as an acetylating agent as well as solvent.
2) Make sure that all salicylic acid is dissolved .
3) The presence of unreacted salicylic acid can be checked by adding ferric chloride solution and observing the color. Formation of intense color indicates yhe presence of unreacted salicylic acid ,in that case recrystallization is must.

EXPERIMENT-4

AIM: TO Prepare a pure and dry sample of Glucosazone

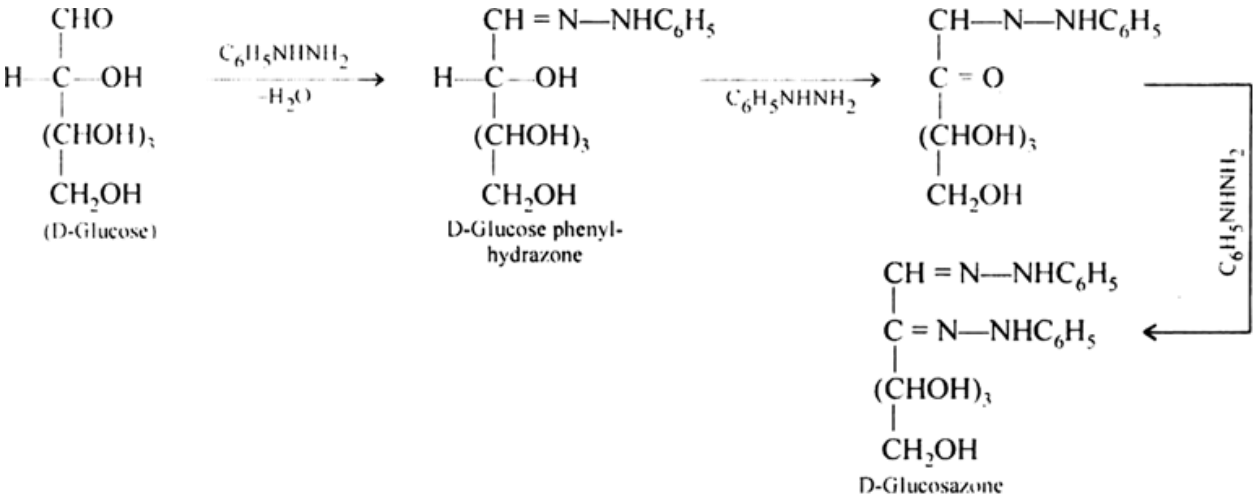
APPARATUS:

Test tube , Funnel , Water bath and measuring cylinder.

CHEMICALS REQUIRED:

Glucose =1gm, Phenyl hydrazine hydrochloride =2 gm ,Sodium acetate=3gm.

REACTION:



PROCEDURE:

1) Take a boiling tube. To this add the given amount of Glucose ,Phenyl hydrazine hydrochloride ,Sodium acetate and 10 ml of H_2O.
2) Mix well by shaking and heat it over water bath for about half an hour .The yellow glucosazone starts separating in about 10 min.
3) Cool the tube by immersing in cold water bath.
4) Filter the ppts and wash it with water and then wih rectified spirit.
5) Dry it in between the folds of filter paper.

c

EXPERIMENT-6

AIM: To Determine The Surface Tension Of An Unknown Liquid By Using Stalagmometer

APPARATUS:

Stalagmometer, Beakers Rubber tubing, Pinch cock, Relative density bottle, Thermometer

THEORY:

When a liquid is allowed to flow through a capillary tube ,a drop is formed at its lower end. It increases to a certain size and falls off. The size of the drop depends upon the radius of the capillary and the surface tension acting along the circumference of the capillary tube supports the drop in the upward direction. The measurement of the surface tension of a liquid is based on the fact that the drop of the liquid at the lower end of the capillary falls down when the weight of the drop becomes equal to the surface tension.

1. Force of gravity exerted on the top = Vdg
   V = volume of the drop
   d = density
   g = gravity
2. The force tending to uphold the drop = $2\pi r.\gamma$
   $2\pi r$ = circumference of the capillary with radius r
   When the two forces are balanced
   $2\pi r.\gamma = Vdg$ ---------- (1)
   n be the number of drops in volume v of the liquid then volume of each drop will be
   $V = v/n$
   Substituting this value in eq (1)
   $2\pi r.\gamma = v/n$ ----------(2)

Consider two liquids of densities d1 and d2 having the surface tensions $\gamma 1$ and $\gamma 2$. Let the number of drops counted for the same volume V of the two liquids be n1 and n2. Then

$2\pi r.\gamma_1 = v/n_1.d_1g$ for first liquid----------(3)

$2\pi r.\gamma_2 = v/ n_2.d_2g$ for second liquid----------(4)

Dividing eq-3 by eq-4, we have

$\gamma_1/\gamma_2 = n_1/n_2 \cdot d_1/d_2$ or $\gamma_1/\gamma_2 = n_1d_1/n_2d_2$

$\gamma_2 = n_1d_2\,\gamma_1/n_2d_1$

where

γ_1 and γ_2 are the surface tension of the two liquids

d_1 and d_2 are the densities of water and solution respectively

n_1 is the no of drops of water

n_2 is no of drops of solution

PROCEDURE:

1) Wash the stalagmometer first and NaOH and then with chromic acid.
2) Immerse the lower end of the stalagmometer in a beaker containing the distilled water. Suck the water until it rises above the mark A, with the help of a screw cock adjust the number of drops to 15 – 20 drops/min
3) Count the number of drops when the liquids falls from A to B
4) Then repeat the experiment with the given liquid whose surface tension is to be measured.
5) Then find the relative densities of the distilled water and the given liquid with the help of relative density bottle.

OBSERVATIONS:

S.No	Distilled water		Unknown Sample	
	No of Drops	Mean no of drops (n1)	No of Drops	Mean no of drops (n2)
1				
2				
3				

CALCULATIONS:

Weight of the relative den sity bottle when empty = w_1g

Weight of the relative density bottle + water = w_2g

Weight of the relative density bottle + given liquid = w_3g

Therefore,

Weight of the water =(w_2- w_1)g

Weight of the liquid = (w_3- w_2)g

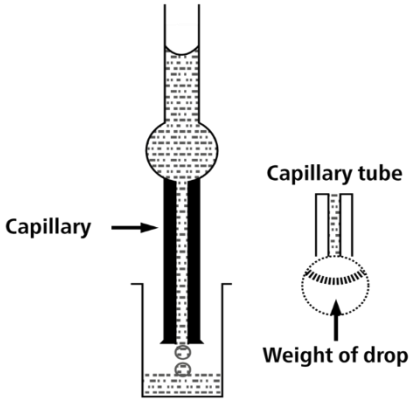
Relative density of liquid = $d_1 / d_w = (w_3 - w_1) / (w_2 - w_1)$

$\gamma_1 / \gamma_w = n_w / n_1 . d_1 / d_w$

where γ_1, d_1, n_1 are the surface tension ,density ,and the number of drops of the given liquid and γ_w, d_w, n_w are the same for water

Surface tension of the liquid =

$\gamma_1 = n_w / n_1 . d_1 / d_w . \gamma_w$ (where γ_w = 72.14 dynes/cm at room temp)



Stalagmometer

PRECAUTIONS:

1) Stalagmometer is highly sensitive to contamination of even minute traces of grease and impurities .It should therefore be thoroughly cleaned with chromic acid.
2) Stalagmometer should be in vertical position throughout the experiment.
3) Drop rate be between 15 -20 drops/min.

EXPERIMENT-7

AIM:TO Determine The Viscosity OF AN UNKNOWN LIQUID BY USING OSTWALD VISCOMETER

APPARATUS:

Oswald viscometer, Stop watch, Specific gravity bottle,10cm^3 pipette, Distilled water, Rubber tubing.

THEORY:

The Ostwald viscometer is based on poiseuille's equation. This relates to the rate of flow of a liquid through a capillary tube with the coefficient of viscosity and is expressed by the equation.

$$\eta = \pi r4tP/8Vl$$

Where v = volume of the liquid

η = viscosity.

t= time

r= radius of the capillary tube

l= length of the capillary tube

P= hydrostatic pressure of the liquid.

Thus,this method involves the measurement of the v t r l and P and id laborious one.Hence a simpler method is used wherein we compare the viscositiwes of the two liquids.If the coefficient of viscosity of one liuid is known,then that of the other can be calculated.

If t_1 and t_2 are the times required to flow for equal volumes of two liquids through the same length of the capilaary tube then from equation 1 we have

$\eta 1 = \pi r4t1P1/8Vl$ ----i

$\eta 2 = \pi r4t2P2/8Vl$------- ii

Divide eq i by Eq ii ,we get

$\eta 1/\eta 2 = P1t1/P2t2$

Now P =hdg =h=height of the liquid,Gravity force,d=Density of the liquid

In case of two liquids h and g are common the formula of viscosity is

$\eta 1/\eta 2 = d1t1/d2t2$

or

$$\eta 2 = \frac{\eta 1\ d2t2}{d1t1}$$

where $\eta 1$ = Viscosity of water

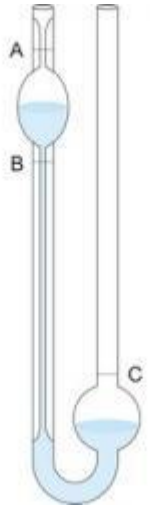
$\eta 2$= Viscosity of unknown sample

d1= Density of water

d2= Density of unknown sample

t1= Flow time of water

t2= Flow time of unknown sample



Ostwalds Viscometer

PROCEDURE:

1. Clean the viscometer with chromic acid and then wash it several times with distilled water. It is finally washed with alcohol and ether and then dried.
2. Attach a piece of clean rubber tube to the end "C" ans clamp the viscometer vertically in the air.
3. Insert a sufficient amount of given liquid in the bulb "B" with the help of pipette the liquid will rise n bulb "A" and bent tube.
4. Now the suck the liquid through rubber tube until it rises above the mark "X" and make sure that there is no air bubble inside the viscometer.

5. Now allow the liquid to fall and start the stopwatchwhen it crosses point"X", and then stop the stop watch when the liquid crosses mark "Y".
6. Repeat the experiment thrice to get concordant readings.
7. Remove the liquid and clean and dry the viscometer again.
8. Repeat the experiment by taking same volume of the distilled water and note the time t_2 taken by the distilled water to pass from X to Y .Repeat it thrice.
9. Weigh the relative denstity bottle when emptythen fill it with given liquid and find its weight.Again fill it with distilled water and find its weight.

OBSERVATION Table

S.No	Distilled water		Unknown Sample	
	Time of Flow (Seconds)	Mean Flow time t1	Time of Flow (Seconds)	Mean Flow time t2

CALCULATION:

Water density at 20^o C ,$d_1 = 0.9982$

Water viscosity $\eta_1 = 1.002$ cP

Density of glycerol (Aq sol) at 20^o C

Glycerol (10%) in water density $d_2 = 1.022$

Glycerol (15%) in water density $d_2 = 1.0345$

Glycerol (20%) in water density $d_2 = 1.0469$

Weight of the relative denisity bottle when empty = w_1g

Weight of the relative density bottle + given liquid = w_2g

Weight of the relative density bottle + distilled water = w_3g

Therefore,

Weight of the liquid =(w_2- w_1)g

Weight of the distilled water = (w_3- w_2)g

Density of liquid / Density of the water = $d_1 / d_2 = (w_2 - w_1) / (w_3 - w_2)$

Relative viscosity of given liquid = $\eta_1/\eta_2 = d_1t_1 / d_2t_2$

Absolute viscosity = $\eta_1 = d_1t_1 /d_2t_2 . \eta_2$

RESULT:

Relative viscosity of the given liquid is _____at –°C

EXPERIMENT-8

AIM: Determine The % Age Of Caco3 In Precipitated Chalk Sample .You Are Provided With 1N HCl And 0.1N NaOH

APPARATUS: Burette,pipette,two beakers (250 ml)Conocal flask 250 ml and burette Stand.

REQUIREMENTS: Chalk sample,Phenolphthalein1N HCl,).1N NaOH

REACTIONS: $CaCO_3 + 2HCl \rightarrow CaCl_2 + H_2O + CO_2 \uparrow$

- 1mole of $CaCO_3$ = 2moles of HCl
- 100gm $CaCO_3$ reactswith2L 1N HCl
- 50 gm $CaCO_3$ reacts with 1L INHCl
- 1gm $CaCO_3$ reacts with 20 ml of 1N HCl

Procedure : To asmall quantity of $CaCO_3$ (ygm) add excess of 1NHCl(30ml0 in aconical flask.To this add 20 ml of H_2O.Let the normality of this solution be N .Now fill the burette with 0.1N NaOH .Pipette out 20 ml solutionacidic chalk solution in a conical flask To it add 1-2 drops of phenolphthalein indicator and titrate it with 0.1 N NaOH filled in burette till light pink colour appears in the flask.Repeat above procedure to get three concordant readings.

Observation Table

S.No	Burette Reading		Volume of 0.1N NaOH used
	Initial Reading	Final Reading	
1			
2			
3			

Concordant reading=_____ml

Calculation: Normality of diluted chalk Sample solution

Chalk solution NaOH

$N_1V_1 = N_2V_2$

N1=Normality of diluted chalk solution

V1= Volume of Chalk solution
N2=Normality of NaOH solution
V2= Vol of NaOH solution used(Burette reading)

$$N1 = \frac{N2 \times V2}{V1}$$

Calculation of unreacted 1NHCl volume

1N HClxV ml = N1x50 ml

$$Vml = \frac{N1 \times 50}{1N} = Xml$$

Volume of 1N HCl reacted with chalk sample = 30 - X = Y ml

1000 ml ,1NHCl reacts with CaCO3=50gm

$$1 \text{ ---------- } = 50/1000 \text{ gm}$$

$$Y \text{ ml } 1NHCl \text{ reacts with } CaCO_3 = \frac{50}{1000} \times Y = Zgm$$

% of CaCO3 in precipitated Chalk sample

A gm of chalk sample contains CaCO3 = Zgm

100gm of chalk sample contain CaCO3= Zx100= % Ans

Precautions:

- All the apparatus should be neat and clean .
- Burette and pipette should be properly filled and no air bubble should be there.
- Reading should be recorded from lower meniscus

EXPERIMENT-9

AIM: To Analyse The Given Antacid Tablets.

APPARATUS : Burette,250 ml Beakers,Measuring flask,titration flask

REQUIREMENT: (Antacid tablets and N/20 HCl),Methyl orange Indicator)

Procedure: Dissolve 9 antacid tablets in water and make up the volume of suspension to 100ml in the measuring flask 100 ml.Transfer the the entire suspension to the the titration flask.Add a few drops of methyl orange to suspension .It turns yellow.Now fill the burette with HCl .Note the initial reading of the burette and titrate the suspension by adding HCl drop by drop with thorough shaking till the yellow colour changes to pink .Note final reading of the burette.
Let the volume of HCl used for 100 ml suspension for its complete neutralization be Vml.

Observation Table

S.No	Burette Readings		Volume of N/20 HCl used
	Initial Reading	Final Reading	
1			
2			
3			

Concordant reading=_____ml

Calculation :

Normality of HCl solution
N1=N/20
Volume of HCl used =Vml(Final –Initial reading)
Volume of suspension (V2)= 100ml
Normality of alkali content antacid tablets N2

$$N2 = \frac{N}{20} \times V = \frac{N2 \times 100}{2000} \times V$$

Amount of OH per litre= N2x17 (Eq Wt of OH =17)

$$= \frac{Vx17}{2000} \text{ gm/L}$$

No of antacid tablets dissolved in 100 ml suspension= 9
No of tablets dissolved per litre = 90

$$\text{Amount of OH per tablets} = \frac{Vx17}{2000} \times \frac{1}{90} \text{ gm}$$

The amount of alkali content Per tablet is =

Precautions :

- All the apparatus should be neat and clean .
- Burette and pipette should be properly filled and no air bubble should be there.
- Reading should be recorded from lower meniscus

EXPERIMENT-10

AIM::Determine volumetrically the % of Cu in a sample of CuSO4 Z gram of which has been dissolved per litre.Provided N/20 $Na_2S_2O_3$

APPARATUS: Burette,250 ml beakers,Titration flask,burette stand .

REACTION : i) $CuSO_4 + 4KI \text{ ------} \square 2CuI_2 + 2K_2SO_4$

$CuI_2 \text{------} \square \quad Cu_2I_2 + I_2$

ii) $2\ Na_2S_2O_3 + I_2 \text{ ------} \square \quad Na_2S_4O_6 + 2\ NaI$

Sodium tetrathionate

Ionic reactions

i) $2Cu^{2+} + 2e \longrightarrow 2Cu^{+}$

$4I^{-} \longrightarrow I_2 + 2I^{-} + 2e$

$2Cu^{2+} + 4I^{-} \longrightarrow 2Cu^{+} + 2I^{-} + I_2$

---

ii) $2S_2O_3^{2-} \longrightarrow 2S_4O_6^{2-} + 2e$

$I_2 + 2e \longrightarrow 2I^{-}$

$2S_2O_3^{2-} + I_2 \longrightarrow 2S_4O_6^{2-} + 2I^{-}$

From the above equation it is evident that

$2Cu^{2+} = I_2 = 2S_2O_3^{2-} = 2e$

PROCEDURE:

Rinse and fill the burette with $Na_2S_2O_3$ .Pipette out of 10 ml of CuSo4 solution in the titration flask.Add 10 ml of 10% KI solution into the flask.Note the initial reading of the burette and titrate the CuSO4 solution with hypo by drop wise addition and constant shaking till the solution acquires light yellow colour.At this point add 1-2 ml of freshly prepared starch solution , a blue color immediately appears in the solution .Continue addition of more sodium thiosulphate till the blue colour just disappears.Note he final reading of the burette.The difference between final and initaial reading gives the volume of thiosulphate solution used..Repeat the titration till three concordant readings are obtained.

Observations :

Normality of CuSO4 solution (N1)= ?
Volume of CuSO4 Solution (V1)=10 ml
Normality of thiosulphate solution (N2)= N/20
Volume of thiosulphate (V2) = (Final – Initial reading) =Vml

Observation Table

S.No	Burette Readings		Volume of N/20 $Na_2S_2O_{3\,(hypo)}$ used (F-I)reading=Vml
	Initial Reading	Final Reading	
1			
2			
3			

Concordant reading = _____ml

Calculation :

Normality of CuSO4 solution N1 =?

Applying normality equation

CuSO4solution Hypo solution

$N_1V_1 = N_2V_2$

$$N_1 \times 10 = \frac{NxV}{20}$$

$$N_1 = \frac{NxV}{20x10}$$

Strength of Copper = 63.5x NxV

---------- gm/litre = Y gm
200

Let impure CuSO4sample is Z gm= 25 gm /litre

Now 25 gm of impure CuSO4 contain pure Copper = Y

Y
1gm impure sample contain pure Copper = -----
25

Y
100 gm impure CuSO4 contains pure Copper = ----- x100 = ----%
25

The percentage of Cupper in CuSO4 sample = ----%

Precautions :

- All the apparatus should be neat and clean .
- Burette and pipette should be properly filled and no air bubble should be there.
- Reading should be recorded from lower meniscus

EXPERIMENT -11

AIM- Compute the percentage of Copper in given brass sample.

APPARATUS: Burette,250 ml beakers,250 ml Titration flask,100 ml Volumetric flask,burette stand

REACTION: (i) $Cu + 4HNO_3 \text{ -----} Cu(NO_3)_2 + 2NO_2 + 2H_2O$

$2\ Cu(NO_3)_2 + 4KI \text{ -----} Cu_2I_2 + I_2 + 4KNO_3$

ii) $2\ Na_2S_2O_3 + I_2 \text{ --} Na_2S_4O_6 + 2\ NaI$

Sodium tetrathionate

Ionic reactions

i) $2Cu^{2+} + 2e \text{ ------} 2Cu^{+}$

$4I^{-} \text{ ------} I_2 + 2I^{-} + 2e$

$2Cu^{2+} + 4I^{-} \text{ ------} 2Cu + 2I^{-} + I_2$

.

ii) $2\ S_2O_3^{2-} \text{ ------} S_4O_6^{2-} + 2e$

$I_2 + 2e \text{ -----} 2I^{-}$

$2\ S_2O_3^{2-} + I_2 \text{ ----} S_4O_6^{2-} + 2I^{-}$

From the above equation it is evident that

$2Cu^{2+} = I_2 = 2\ S_2O_3^{2-} = 2e$

PROCEDURE-Weigh 0.5 gm brass sample and transfer it into 250 ml conical flask.Add 10 ml 1:1 HNO3 acid and heat it to 85^0 C till brass sample dissolves .Add 1gm urea to it to

destroy oxides of Nitrogen .warm it for few minutesand transfer the contents to 100 ml volumetric flask and make up the volume by adding distilled water.Pipette out 20 ml of this solution into titration flask .To it add liquor ammonia solution till bluish white ppts are formed .To it add 10 ml acetic acid and 20 mlk of 10%KI solution .The colour of the solution will be dark brown.Titrate it with Sodium thiosulphate(hypo) solution taken in burette till pale yellow colour is obtained. Stop addition of hypo and add 1-2 ml of 1% starch solution to theflask contents.A dark blue coloured solution is obtained.Resume the addition of hypo till blue colour disappears.Note the final reading on burette.Final minus initial reading givesthe volume of the hypo consumed . Repeat the procedure with fresh aliquots and note the volume of hypo consumed.Note the concordant reading. Let it be x ml

Observation table

S.No	Volume of Brass solution (ml)	Burette reading		Vol of Hypo used (ml)
		Initial Reading	Final reading	
1	20			
2	20			
3	20			
4	20			

x= …ml

Calculation

Normality of Brass solution

Brass Sol Hypo
N1V1 = N2V2
= N2xV2/V1=N1
Strength of Cu= 63,5x N1= gm /L=Z

W gm brass contain Cu= Z
100 gm brass = Zx100/w

Result== Cu %

Precautions :

- All the apparatus should be neat and clean .
- Burette and pipette should be properly filled and no air bubble should be there.
- Reading should be recorded from lower meniscus

Experiment 12

AIM: Determine the dissociation constant of weak acid (Acetic acid) by pH meter.

INTRODUCTION: In this experiment you are going to perform a titration of a weak acid against a strong base and we measure pH changes during titration by pH meter. The pKa value of acetic acid is determined by plotting pH titration curve.

The pH at the half neutralization of a weak acid can be determined by measuring the pH with a pH meter after each addition of a strong base and creating a titration curve which resembles the one shown in the Fig. 2.1 below. Once the equivalence has been determined, the pH can be read halfway to the equivalence point, and from it the Ka of the acid can be calculated.

REQUIREMENTS

Apparatus pH meter with combination pH electrode

Chemicals Oxalic acid solution, 0.10 M NaOH solution, 0.10 CH3CHOOH solution.

PROCEDURE

\ **Part A:** Standardization of NaOH and Acetic acid. 1. Prepare oxalic acid solution (0.1 M) by dissolving appropriate amount of oxalic acid to 100 cm3 of distilled water in a volumetric flask of 100 cm3 . Using this solution as primary standard, standardize NaOH solution (0.1N) using phenolphthalein indicator. Report your observations in the observation Table 1.

2. Similarly, using above NaOH solution as secondary standard, standardize given acetic acid solution. Report your observations in the observation Table 2.

Part B: Determination of the Ka of Acetic Acid. 1. Perform a titration by taking 10 cm3 acetic acid against NaOH using phenolphthalein indicator to know the position of end point. 2. Again take 25 cm3 acetic acid in a 100 cm3 beaker add 20-25 cm3 of distilled water and dip the pH electrode, find the initial pH using pH meter (you pH meter should be pre-

calibrated with standard buffers). Add standardized NaOH from the burette in small instalments of 1-2 cm3 and stir well. Record the pH after each addition. Near the end point, NaOH should be added in very small instalments of 0.1-0.5 cm3 . At the end point, you will observe sudden change in the pH and continue the titration until pH readings remain relatively constant at a pH of 10- 12. Record your observation in the observation Table 3. Your pH reading should provide a smooth titration curve. If not, try again. 3. Plot a graph of pH (y-axis) vs. volume NaOH added (x-axis). Determine the equivalence point from the graph. Find out the NaOH volume used in complete neutralization of acetic acid. From plotted curve find out the cm3 of NaOH and pH at ½ at the neutralization of acetic acid. Calculate the value of Ka using Eq. pKa = pH.

Observation –I

Standardization of NaOH

S.No	Volume of oxalic acid (ml)	Volume of NaOH (ml)
	10	
	10	
	10	

Volume of NaOH used for end poiunt=xml

Normality of NaOH using normality equation=N1V1=N2V2=N1

Observation -2

Standardization of Acetic acid

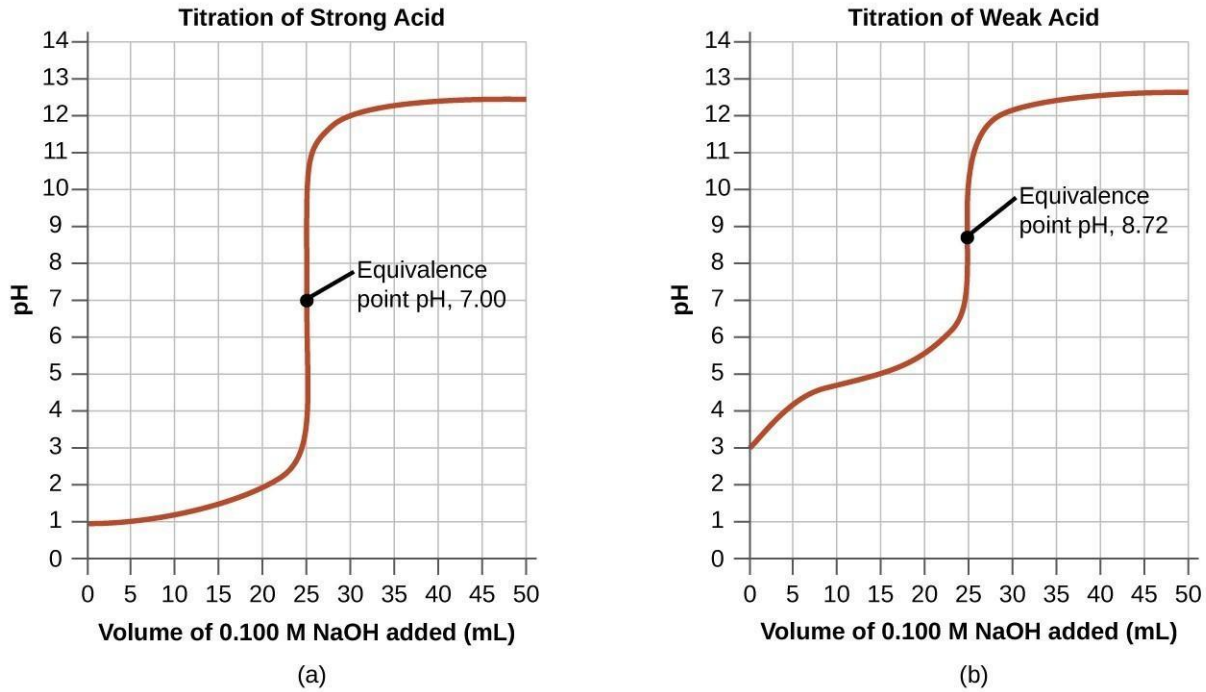
S.No	Volume of Acetic acid (ml)	Volume of NaOH (ml)
	10	
	10	
	10	

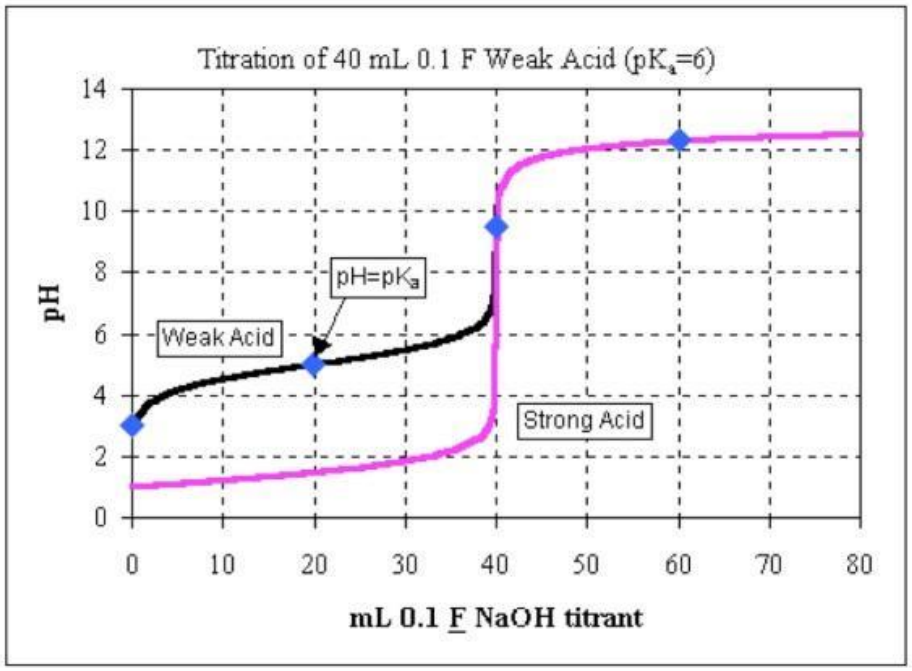
Normality of Acetic acid=N1

Observation -3 : Determination of the Ka of CH3COOH

S.No	Vol of NaOH(ml) Added	pH
1	00	
2	2	
3	4	
4	6	
5	8	
6	10	
7	12	
8	14	
9	16	
10	18	
11	20	
12	22	

RESULT: Ka value from half neutralization method = Henderson-Hasselbalch equation, **pH=pKa at the half-equivalence point**





0.1F =0.1M

Figures- Dissociate constant of weak acid by pH meter

PRECAUTIONS

• Never touch the membrane of the glass electrode with anything else except soft tissue paper since it is fragile and is easily ruined if scratched or bumped.

• The electrode(s) must not be removed from the solution unless the selector switch is at zero.

• Before measuring pH, establish equilibrium between electrodes and titration solution by stirring solution to ensure homogeneity

EXPERIMENT 13

AIM:Estimate the acids in acid mixture conductometrically

INTRODUCTION : In this experiment you are going to perform a conductometric titration of a mixture of a strong acid and weak acid with a strong base. Conductance changes during the titration are measured by conductometer. The equivalence points of the titration are detected by plotting a titration curve using conductance values and volumes of a base added.

In this curve there are two break points. The first break point corresponds to the neutralization of strong acid. When the strong acid has been completely neutralized only then the weak acid starts neutralizing. The second break point corresponds to the neutralization of weak acid and after that the conductance increases due to the excess of OH- ions in case of a strong base as the titrant. However, when the titrant is a weak base, it remains almost constant after the end point.

Chemicals Sodium hydroxide, Hydrochloric acid and acetic acid. Solution provided 1. 0.1 M Sodium hydroxide solution which was standardized with standard oxalic acid. 2. Mixture of ~ 0.01 M HCI (15 cm3) and ~ 0.01 M acetic acid (15 cm3)

PROCEDURE . Take 30 ml of given solution[mixture of ~ 0.01 M HCI (15 ml) and ~ 0.01 M acetic acid (15 ml)]in a 50 ml beaker and dip the conductance cell into it. 2. Take NaOH solution in the burette. 3. Connect the conductometer to the mains and to the conductance cell. Switch on the instrument keeping the meter switch at 'CAL'. 4. Calibrate the meter keeping the selector knob at " 20 mS' by rotating the sensitivity knob till the meter reads 1.0. 5. Shift the meter switch to 'READ'. Read the conductance of the solution. Record this value in observation Table 4.3. 6. Make additions of NaOH from the burette as given in Observation Table 1. After each addition, stir the solution well and read the conductance. Enter all the conductance values in observation Table 7. Plot conductance versus volume of NaOH on a graph sheet and calculate the volume of NaOH used for the neutralization of HCl and acetic acid respectively.

Observation Table-1

Volume of NaOH added (ml)	Conductance (mS)
00	
0.3	
0.6	
0.9	
1.2	
1.80	
2.10	
2.40	
2.70	
3.30	
3.60	
3.90	
4.20	
4.50	
5.10	

CALCULATIONS Volume of the acid mixture taken = 30 cm3 [mixture of ~ 0.01 M HCI (15 cm3) and ~ 0.01 M acetic acid (15 cm3)] Molarity of standardized NaOH solution = M 1 mol dm-3 = …….. mol dm-3 . 1. From the graph, the first change in slope of the conductometric curve gives the first equivalence point (obtained after complete neutralisation of HCI present in the acid mixture). From which volume of NaOH corresponding to this first equivalence point V1 cm3 can be determined. 2. Similarly, the volume of NaOH, corresponding to the second equivalence point V2 cm3 (total volume of NaOH used for the complete neutralization of both the acids.

Result

Molarity of HCI = M1V1/15 mol dm-3 …… mol dm-3 Molarity of CH3COOH = M1 (V2 – V1)/15 mol dm-3 ……mol dm-3

Precautions

1. After switching on the instrument (conductometer), it should be allowed to stabilize prior starting the experiment.

2. The conductance cell must always be dipped either in solution or in distilled water.

3. The platinum electrodes of the conductance cell must be completely immersed in the solution during the measurement of conductance.

4. There should be no air bubble between the two electrodes. 5. The titrant must be at least ten times more concentrate than the analyte

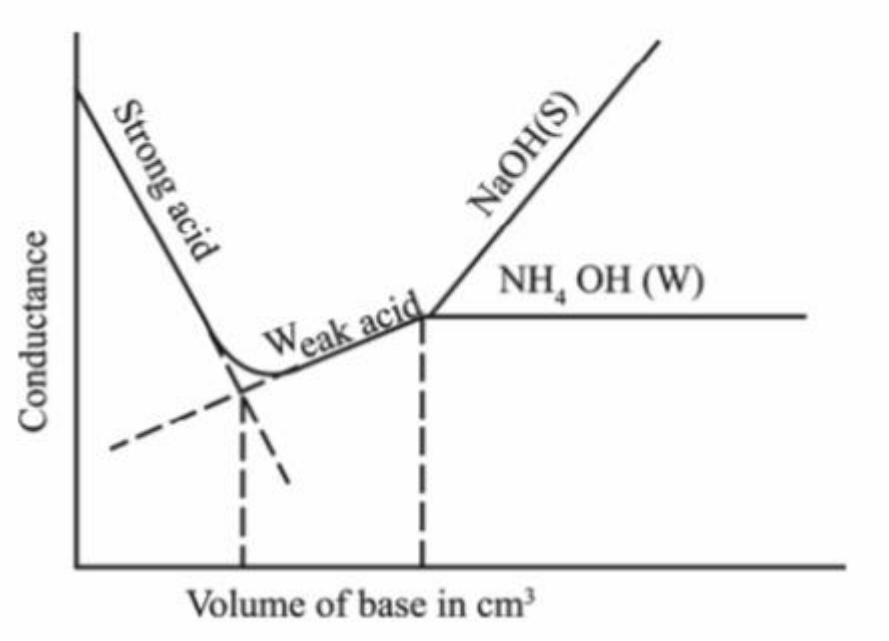


Figure- Conductance Titration Curves

Experiment14

AIM-To determine the chemical oxygen demand of waste water sample using Potassium Dichromate(K2Cr2O7)

Reaction : Organic matter + $Cr_2O_7^{2-}$ + H^+ + (acid) → Cr^{3+} + CO_2 + H_2O

$$\text{Oxidant } Cr^{6+} \xrightarrow{Reduction} Cr^{3+}$$

$$\text{Organic Matter} \rightarrow CO_2 + H_2O$$

INTRODUCTION

Chemical oxygen demand (COD) is the measure of oxygen required in oxidising the organic compounds present in water by means of chemical reactions involving oxidizing substances such as potassium dichromate and potassium permanganate. Potassium dichromate is the most suitable oxidant but for waters having more than 2 gll of chlorides potassium permanganate is used. The estimation of COD is of great importance for waters having unfavourable conditions for the growth of microorganisms, such as presence of toxic chemicals. In such water BOD cannot be determined accurately. COD is always higher than BOD, by approximately 2 to 3 times of BOD.

Reagents

(1) Potassium Dichromate Solution (0.25 N) K2Cr207 Dissolve 12.258 g of K2Cr207 in the distilled water and dilute it to 1000 ml.
(2) (2) Silver Sulphate (Ag2SOi) Dry Powder The purpose of adding silver sulphate powder is to avoid interference from straight c chain aliphatic and aromatic compounds.
(3) (3) Mercuric Sulphate (HgS04) Dry Power It is added to water sample to avoid interference from chlorides. 10 mg of HgS04 is required per 1 mg of chloride.
(4) (4) Sulphuric Acid (&So4) Concentrated It is used for acidification of water sample.
(5) (5) Ferroin Indicator Solution Mix and dissolve 1.485 g of 1, 10-phenanthroline monohydrate together with 695 mg of FeS04 . 7H20 in the distilled water and dilute it to 1000 ml of solution. (6) Ferrous Ammonium Sulphate Solution (0.25 N Fe (NH4)2 (SO4)2.

(6) 6H20) Dissolve 98.026 g of ferrous ammonium sulphate in the distilled water and after adding 20 ml of concentrated H2SO4, dilute it to make 1000 ml of solution.

PROCEDURE

Take 20 rnl of sample in the flask and add 10 ml of Potassium dichromate solution, a pinch of each silver sulphate and mercuric sulphate and 30 ml of sulphuric acid. Attach condensor to the mouth of flask and heat the flask on a hot water bath or heating mental for at least two hours to reflux the contents. Cool the flask, detach from unit and dilute its contents to 150 ml by adding distilled water. Add 2-3 drops of Ferroin indicator solution and titrate'against ferrous ammonium sulphate solution. At the end point blue green colour of contents changes to reddish blue. A blank sample of distilled water is carried through the same COD testing procedure as the waste water sample. The purpose of running a blank is to compensate for any error that may result because of the presence extraneous organic matter in the reagents.

Observations

(i) Sample Titration

S.No	Volume of Sample Solution taken	Burette Reading		Vol of FAN solution Used(ml)
		Initial	Final	
1				
2				
3				
4				

(ii) Blank Titration

S.No	Volume of distilled water taken taken	Burette Reading		Vol of FAN solution Used(ml)
		Initial	Final	
1				
2				
3				
4				

Concordant Reading =…ml

CALCULATIONS

COD mg/l =(B – A) x N x1000 x 8/V

where, A = Volume of titrant used against sample (in ml),

B = Volume of titrant used against Blank (in ml),

N = Normality of titrant (0.25N), and

V = Volume of Sample taken (in ml).

Precautions

- All the apparatus should be neat and clean .
- Burette and pipette should be properly filled and no air bubble should be there.
- Reading should be recorded from lower meniscus