SRI INDU INSTITUTE OF ENGINEERING AND TECHNOLOGY

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(Approved by AICTE, New Delhi and Affiliated to JNTUH, Hyderabad)
KhalsaIbrahimpatnam, Sheriguda(V), Ibrahimpatnam(M), Ranga Reddy Dist., Telangana – 501510

DEPARTMENT OF HUMANITIES AND SCIENCES

ENGINEERING CHEMISTRY LAB
MASTER MANUAL

(R18-IB.Tech)

<u>CERTIFICATE</u>

*ThisistoCertifyThat***PreparedbyDr.Premalatha,G.Vijay**

V.Swapna,K.Mounika,K.Sharanya,*have preparedmanualof*

practical work done in the Engineering Chemistry laboratory ,.

LabIncharge   H.O.D

INDEX

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JNTUH (R18) B.Tech-I Syllabus

CH106BS/CH206ES:ENGINEERINGCHEMISTRYLAB

</div>

B.Tech.I YearI Sem   **LTP   C**

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ListofExperiments:

1. Determinationoftotalhardnessofwaterbycomplexometricmethodusing EDTA
2. Determinationof chloride contentofwaterbyArgentometry
3. EstimationofanHClbyConductometrictitrations
4. EstimationofAcetic acidbyConductometric titrations
5. EstimationofHClbyPotentiometrictitrations
6. EstimationofFe^{2+}byPotentiometryusingKMnO$_4$
7. Determinationofrateconstantofacidcatalysedhydrolysis ofmethylacetate
8. SynthesisofAspirinandParacetamol
9. Thin layer chromatography calculation of R$_f$ values. eg ortho and para nitrophenols
10. Determinationofacidvalueofcoconutoil
11. VerificationofFreundlichadsorptionisotherm-adsorptionofaceticacidon charcoal
12. Determination of viscosity ofcastor oil and ground nut oil by using Ostwald's viscometer.
13. Determinationofpartitioncoefficientofaceticacidbetweenn-butanoland water.
14. DeterminationofsurfacetensionofagiveliquidusingStalagmometer.

References

1. Seniorpracticalphysicalchemistry,B.D.Khosla, A.GulatiandV.Garg (R. Chand & Co., Delhi)
2. Anintroduction to practicalchemistry,K.K.SharmaandD.S.Sharma (Vikas publishing, N. Delhi)
3. Vogel'stextbookofpracticalorganicchemistry5thedition
4. TextbookonExperimentsandcalculationsin Engineeringchemistry–S.S. Dara

SRIINDUINSTITUTEOFENGINEERING&TECHNOLOGY

ENGINEERINGCHEMISTRYLAB

LIST OF EXPERIMENTS:

1. Determinationoftotalhardnessofwaterbycomplexometricmethodusing EDTA.
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12. Determination of viscosity ofcastor oil and ground nut oil by using Ostwald's viscometer.
13. Determinationofpartitioncoefficientofaceticacidbetweenn-Butanoland water.
14. DeterminationofsurfacetensionofagivenliquidusingStalagmometer.

ADDITIONALEXPERIMENTS:

15. EstimationofMohr'ssaltusingstandardKMnO$_4$solution.
16. DeterminationofsaltconcentrationbyIon- exchangeresin.

References

1. Vogel'stextbookofpracticalorganicchemistry5thedition
2. TextbookonExperimentsandcalculationsin Engineeringchemistry–S.S. Dara

COURSEOBJECTIVES*:*

1. Tobringadaptabilitytotheconceptsofchemistryandtoacquiretherequiredskillsto become a perfect engineer.

2. Toimpart thebasic knowledge ofatomic, molecular andelectronicmodifications which makes the student to understand the technology based on them.

3. Toemphasizeonthetypesofhardnessofwater,disadvantagesofusinghardwaterin industries and necessity to soften hard water by various modes.

4. Toacquiretheknowledgeofelectrochemistry,corrosionandwatertreatmentwhichare essential for the engineers and in industry.

5. Toacquiretheskillspertainingtospectroscopyand to applythem for medical andother fields.

6. To impartthe knowledge of stereochemistry and synthetic aspects usefulfor understanding reaction pathways.

COURSEOUTCOMES:

C116.1.Studentisabletoacquirethescientificattitudebymeansofdistinguishing,analyzing and
solvingvarious engineering problems.

C116.2.Studentisable togainthe knowledge ofatomic,molecularand electronicchanges,band
theory related to conductivity.

C116.3.Studentisabletodifferentiatebetweenhard&softwater,theireffectswhenused in
Thermal power plants and various methods involved in water softening.

C116.4.StudentisabletoapplytheprinciplesandconceptsofElectrochemistry,corrosionand
mechanism associated with corrosion control methods to produce advanced corrosion
resistant materials like PVC, PS.

C116.5.Studentisabletoapplytheconceptofbasicspectroscopytomedicalandother fields.

C116.6.Studentisabletoacquiretheknowledgeofconfigurationalandconformationalanalysis of
molecules andreaction mechanisms.

InstructionsforRecordWriting

1. Writeontherighthandpagethefollowinginorder:
   a) Serialnumberanddateofperformance(inthemargin)
   b) Nameandnumberoftheexperimentasgiveninthelist
   c) Aimofthe experiment
   d) Descriptionoftheapparatus
   e) Procedureincludingsourcesoferrorandprecautionstakentoeliminateorto minimize them.
   f) InferenceorResult.
   g) Explanation,ifnecessaryofanydivergenceintheexpectedresult.

2. Lefthandpageshouldcontainthefollowingintheirproperplaces.
   a) Neatdiagramofthemainapparatusused
   b) Observationintabularform
   c) Calculationintabularform
   d) Graphsheetsandotherpaperstobeattached.

N.B.(a)Studentsshouldsubmitarecordofthepreviousexperimentswhenthey comefor practical work.

(b) Anexperimentisdeemedtobecompletewhenitissatisfactorilyperformed and recorded.

(c) Allsuggestionsgivenordefectsnotedshouldbeattendedtowhentherecords are taken back.

KEEPTHERECORDBOOKNEATITFETCHESMARKS

EXPERIMENTS

Experiment-1

DeterminationofTotalHardnessofWaterbyComplexometric Method Using EDTA
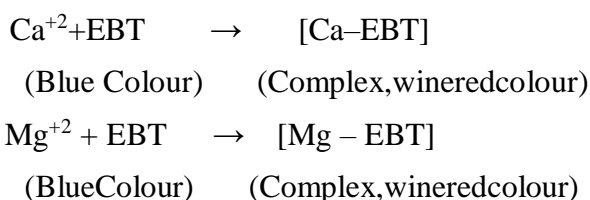
Objective: Toestimate thetotalhardnessofwaterbyEDTAMethod.

Outcome: Thestudentswillbeable toanalysethenatureofsaltscausinghardnessandto solve the engineering problems arising during steam production in Boilers.

ChemicalsRequired: Ammoniabuffersolution,EriochromeBlack–T(EBT),Eriochrome Black – T (EBT), Magnesium chloride, EDTA Solution, Hard water

Apparatus: ConicalFlask,Pipette,Burette,Beaker

Principle:
Ethylene diammine tetra acetic acid (EDTA) forms stable complexes with $Ca^{+2}$and Mg^{+2} ions present in water at pH 9-10. The sample of hard water must be treated with ammoncal buffersolution andEBT– indicatorwhichformsunstable, winered colouredcomplex with Ca^{+2} and Mg^{+2} present in water.

$$Ca^{+2}+EBT \quad \rightarrow \quad [Ca–EBT]$$

(Blue Colour)   (Complex,wineredcolour)

$$Mg^{+2} + EBT \quad \rightarrow \quad [Mg – EBT]$$

(BlueColour)   (Complex,wineredcolour)

Thestabilityof ametalindicatorcomplexislessthanthatofmetalEDTAcomplex.During thetitrationofthe complexwithEDTA,EDTAextractsthemetalionsfromthemetal –ion – EBT indicator complex and forms stable, colorless complexes by releasing the free indicator. Where by the end point of the titration is the colour change from wine red to blue.

$[(Ca^{+2}–EBT) +(Mg –EBT)] +EDTA \rightarrow (Ca– EDTA)+ (Mg – EDTA)]+EBT$
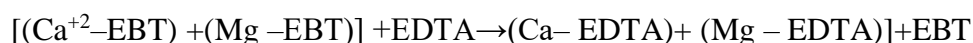
Wine red coloured complex   Colorless&stablecomplex   Bluein
            Colour

Procedure:

Preparationofstandardhardwater*:*

Dissolveonegramof pure,dry $CaCO_3$ inminimum quantity ofdiluted HCl andevaporatethe solution to dryness on water bath. Dissolve the residue in small amount of water and transfer intoa100mlstandardflask.Makeupthesolutiontothemarkwithdistilledwaterandshakethe   flask well for uniform concentration.

Standardization ofEDTA Solution:

Pipetteout10mlofstandardhardwatersolutionintoaconicalflask,add2mlofbuffersolution   and2-3dropsofEBTindicatorandtitratethewineredcolouredcomplexwithEDTAsolution takenintheburette,afterrinsingtheburettewithEDTAsolution,tillthewineredcolourofthe   solution changes to blue colour. Note the burette reading repeat the titration to get concurrent values.

Estimationofhardness ofsamplewater:

Pipette out2 ml ofthewatersample into a25ml conical flask, add2mlofbuffersolutionand 3 drops of EBT – indicator. Titrate the wine red coloured solution with EDTA taken in the burette, till a clear blue coloured end point is obtained. Repeat the titration to get concurrent values.

Observation& Calculations

StandardizationofEDTA:

S.No.	Volume of hard water	Burettereading		Volume of EDTA
		Initial	Final	
1				
2				
3				

EstimationofHardness:

S.No.	Volumeof Sample hard water	Burettereading		Volume of EDTA
		Initial	Final	
1				
2				
3				

MolorityofstandardHardwaterCaCO$_3$

$$M_1 = \frac{Wt\ of\ CaCO_3}{M.wt\ of\ CaCO_3} X \frac{1}{Volume\ of\ solution}$$

$$= \underline{\qquad} M$$

MolarityofEDTA Solution(M$_2$)=?

$$\frac{V1M1}{n1} = \frac{V2M2}{n2}$$

$$n_1 = n_2 = 1$$

$M_2 = \dfrac{V1M1}{V2} = \underline{\qquad} M$

V_1 = Volumeofstandardhardwater = $\underline{\qquad}$ ml

V_1 = VolumeofEDTA = $\underline{\qquad}$ ml

M_1 = Molarityofstandardhard water = $\underline{\qquad}$ M

M_2 = MolarityofEDTA = $\underline{\qquad}$ M

Molarityof thesampl ewat er= $\underline{\qquad}$ (M$_3$) or(Msampl e)

M_2V_2 = M_3V_3

M_3 = $\dfrac{M2V2}{V3}$ = $\underline{\qquad}$ M

V_2 = volumeofEDTA

M_2 = MolarityofEDTA

V_3 = volumeofhardwater

Totalhardnessofwater = M$_3$ x 100 x1000

$$= \underline{\qquad} ppm.$$

Result:

Thetotalhardness of sampleof water = $\underline{\qquad}$ ppm.

Experiment-2

Determinationofchloridecontentofwaterby Argentometry

Objective: TodeterminechlorideionconcentrationinawatersamplebyArgentometry (Mohr's method).

Outcome: Thestudentshallbeabletoidentify &estimatetheamountofchlorideions present in given water sample.

**Chemicalsrequired:**Silvernitrate($AgNO_3$),NaCl,Potassiumchromate(K_2CrO_4) indicator.

Apparatus: Beaker,burette,pipette,conicalflask,measuringjar,volumetricflask

Principle:

Chloride ion (Cl^-) is one of the major inorganic anions in water and wastewater. Along the seacoastchloridemaybepresentinhighconcentrationbecauseofleakageofsaltwaterinto thesewagesystem. Italso may beincreased by industrialprocess. A high chloridecontents may harm metallic pipes and structures as well as growing plants. The measured chloride ionscanbeusedtoknowsalinityofdifferentwatersources.Forbrackishwater(orseawater orindustrialbrinesolution),itisanimportantparameterandindicatestheextentofdesalting ofapparatusrequired. Generally theMohr'smethod isusedto estimatethechloridepresent in water sample.

The Mohr method uses chromate ions as an indicator in the titration of chloride ions with a silver nitrate standard solution. After all the chloride has been precipitated as white silver chloride, the first excess of titrant results in the formation of a silver chromate precipitate, which signals the end point (1).

Thereactionsare:

$$Ag^+ + Cl^- \leftrightarrow AgCl(s)$$

$$2Ag^+ + CrO_4{}^{2-} \leftrightarrow Ag_2CrO_4(s)$$
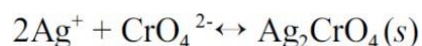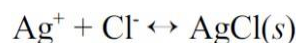
Procedure:

Preparationof 5% K_2CrO_4 (indicator): Dissolve 1.0 g of K_2CrO_4 in 20 ml of distilledwater.

Preparation of standard $AgNO_3$ solution: Transfer 9 g of $AgNO_3$ to a 500 ml volumetric flaskandmadeuptovolumewithdistilledwater.Theresultingsolutionwas approximately 0.1 M.

Standardization of $AgNO_3$: Transfer 0.2500 g portions of NaCl into conical flask and dissolved in about 100 mL of distilled water. In order to adjust the pH of the solutions, add smallquantitiesof$NaHCO_3$untileffervescenceceased.Addabout2mlofK_2CrO_4andtitrate the solution to the first permanent appearance of red $Ag_2Cr_2O_4$.

DeterminationofCl^- insolidsample: Take20 ml ofunknownwatersample in to conical. Addsmallquantitiesof$NaHCO_3$untileffervescenceceased.Addabout2mlof$K_2CrO_4$and titrate the solution to the first permanent appearance of red Ag_2CrO_4.

Interpretationof data

StandardizationofAgNO$_3$:

Replicate	Weightof NaCl(gr)	VolumeofAgNO3 (ml)	Concentrationof AgNO$_3$ (M)
1	0.25	Vml	M$_1$
2	0.25	Vml	M$_2$
3	0.25	Vml	M$_3$

CalculationsforReplicate1of standardization:

MolecularmassofNaCl=58.44 g/mole

$$MmolesofAgNO_3 = \frac{0.25 \text{ gr NaCl}}{58.44 \text{ gr/mole}} \times \frac{1000 \text{ m moles NaCl}}{1 \text{ mole NaCl}} = 4.278 \text{mmoles Molarity of}$$

$$AgNO_3 = \frac{4.278}{V \text{ ml AgNO3}} = M$$

$$\text{Molarityof } AgNO_3 = \frac{M1+M2+M3}{3} = M$$

DeterminationofChloridein Unknown:

Replicate	Volumeofwater sample	VolumeofAgNO3 (ml)	ConcentrationofCl$^-$ solution
1	20 ml	Vml	
2	20 ml	Vml	
3	20 ml	Vml	

Atomicmassof Cl$^-$= 35.45g/mole

$$MmolesofCl^- = M_{AgNO3} X V_{AgNO3} = MXV = x Mmoles \text{ Mass of Cl}^-$$

= x M moles X 35.45 = y mg

Experiment-3

EstimationofanHClbyConductometricTitrations

ObjectiveTodeterminethestrengthofthestrongacidbytitrationwithstrongbaseConductometr ically.

**Outcome:**Thestudentshallbeabletoanalyzethevariationofconductancevaluesofgiven acidwith addition of strong base using conductivity meter .

ChemicalsRequired: Sodiumhydroxide,Hydrochloricacid

Apparatus: ConductivityBridge,Conductivitycell,Burette,Beakers,Standardflask, pipette, Burette Stand.

Principle:
At first solution contain H+ and Cl⁻ ions. Since H⁺ions posses greater mobility it follows that the conductivity is mainly due to H+ ions. The addition of NaOH is represented by the equation.

$$H^+ + Cl^- + Na^+ + OH^- \longrightarrow Na^+ + Cl^- + H_2O$$
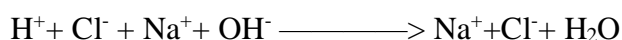
As NaOH is added the H^+ ions are removed. The conductivity decreases as Na^+ ions do not process much mobility. As the neutralization point and solutions contains Na^+ions and Cl^- ionsandwillhaveminimumconductancevalue.IfNaOHisfurtheraddedthiswilladdOH⁻ ions and so the conductivity increases.

Procedure:
A standard solution of 1M NaOH is prepared. Similarly, 0.1M HCl is prepared. 200 ml of HClistakenina250mlbeaker.Further,theconductivitycelliswashedwithdistilledwater andrinsedwithacidsoln.Thecelliskeptinacidcontainingbeakeranditisconnectedtothe bridge.Theconductivityofthesolutionismeasuredbyadjustingthereading.NaOHsolution is taken into burette and add 1 ml of solution to acid, stirred well and conductance is measured. Each time 1 ml of base is added to acid stirred well and the conductance is measured. For every instance equal numbers of values are taken on either side of the point ofmaximum.Repeattheprocedureofadditionof1mlNaOHandnotingtheconductivityof the resulting solution. Take 20-25 readings
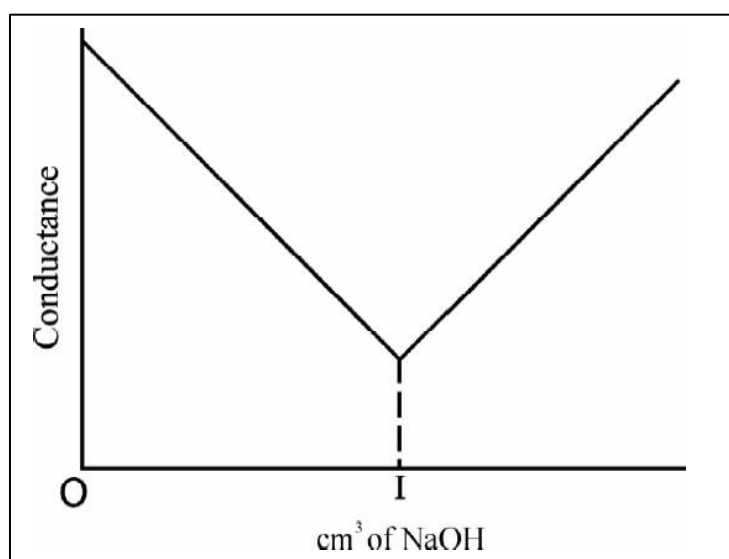
Calculations:

Volumeofunknownacid(V_1)=200 ml.

S.No	Volumeof NaOH(mL)	Observedconductance
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		
20		
21		
22		

FORMULA:

$$N_1V_1(HCl)=N_2V_2(NaOH)$$

$$N_1= \frac{1 \times V_2}{200}$$

Strengthofsolution=Eq.WeightX Normality

$$= 36.5 \times N_1$$

$$= - - - - - - - - - - \text{ g/lit}$$

RESULT:

TheNormalityofstrongacid(HCl)determinedbytitratingagainstastrongbase(NaOH)
=_____N

15

Precautions:

1. Theconductivitycellshouldbehandledverycarefullyasitisvery delicate.

2. Stirringshouldbedone aftereachadditionofthetitrant.

Advantagesorapplications ofconductancetitration:

1. Thismethodcanbeusedtoverydilutesolutions.

2. Givesveryaccurateendpointswithanerrorof±0.5

3. These titrations are very useful in case of coloured solutions which cannot be titrated by ordinary volumetric method because colour change of indicator is not clear.

4. Usefuloftitrating weakacidsagainstweakbases,whichotherwisedonotgivesharpend points.

5. Nokeenobservationisnecessaryneartheendpointsinceitisdetectedgraphically.

TheNormalityofstrongacid(HCl)determinedbytitratingagainstastrongbase(NaOH)
=_____N

Experiment-4

EstimationofAceticAcidbyConductometricTitrations

Objective: TodeterminethestrengthoftheweakacidbytitrationwithstrongbaseConductometrically.

**Outcome:**Thestudentshallbeabletoanalyzethevariationofconductancevaluesofgiven weak acidwith addition of strong base using conductivity meter.

ChemicalsRequired: Sodiumhydroxide,aceticacid

Apparatus: ConductivityBridge,Conductivitycell,Burette,Beakers,Standardflask, pipette, Burette Stand.

Principle:
Before titration low initial conductance is observed due to low H^+ obtained during dissociation of weak CH_3COOH. During titration we can observe slight decrease of conductance due to consumption of H^+. During progress of titration we can observe slight increase in conductancedue to the presence of $CH3COO^-$&Na^+ and nearly constant H^+ due to the buffer action of the produced CH_3COONa and the remaining CH_3COOH. After end point excess NaOH will lead to increase in conductance due to increasing of $Na^+$and OH^-.

$$CH_3COOH+Na^++ OH^-\rightarrow CH_3COO^-+Na^++H_2O$$
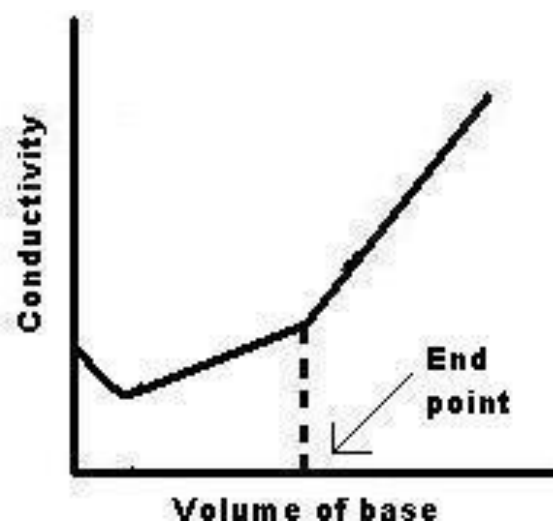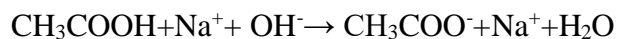
Procedure:
Prepare a standard solution of 1M NaOH. Similarly, prepare 0.1M acetic acid. 200 ml of aceticacidistakenina250mlbeaker.Further,theconductivitycelliswashedwithdistilled waterandrinsedwithacidsoln.Thecelliskeptinacidcontainingbeakeranditisconnected to the bridge. The conductivity of the solution is measured by adjusting the reading. NaOH solution is taken into burette and add 1 ml of solution to acid, stirred well and conductance is measured. Each time 1 ml of base is added to acid stirred well and the conductance is measured. For every instance equal numbers of values are taken on either side of the point ofmaximum.Repeattheprocedureofadditionof1mlNaOHandnotingtheconductivityof the resulting solution. Take 20-25 readings

CALCULATIONS:

Volumeofunknownacid(V_1)=200 ml.

S.No	Volumeof NaOH	Observedconductance
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
11		
12		
13		
14		
15		
16		
17		
18		
19		
20		

FORMULA:

$$N_1V_1(CH_3COOH) = N_2V_2(NaOH)$$

$$N_1 = \frac{1 \times V_2}{200}$$

Strengthofsolution=Eq.WeightX Normality

$$= 36.5 \times N_1$$

$$= \text{- - - - - - - - - -} \ g/lit$$

RESULT:

Thenormalityofweakacid(aceticacid)determinedbytitratingagainstastrongbase (NaOH) =_N

Precautions:

1. Theconductivitycellshouldbehandledverycarefullyasitisvery delicate.

2. Stirringshouldbedone aftereachadditionofthetitrant.

Advantagesorapplications ofconductancetitration:

1. Thismethodcanbeusedtoverydilutesolutions.

2. Givesveryaccurateendpointswithanerrorof±0.5

3. These titrations are very useful in case of coloured solutions which cannot be titrated by ordinary volumetric method because colour change of indicator is not clear.

4. Usefuloftitrating weakacidsagainstweakbases,whichotherwisedonotgivesharpend points.

5. Nokeenobservationisnecessaryneartheendpointsinceitisdetectedgraphically.

Experiment-5

EstimationofHClbyPotentiometry

Objective: To determine the equivalencepoint between strong acid and strong base and to determine the normality of HCl by titrating with NaOH using potentiometer.

Outcome : The student shall be able to analyze the variation of EMF values of given acid with addition of strong base using potentiometer.

Chemicalsrequired:0.1NHCl,1NNaOH,distilledwater.

Apparatus: Potentiometer, standard cell, saturated calomel electrode, platinum electrode, beaker, burette, stirrer etc.

Principle
When a solution of strong acid (HCl) is titrated with the solution of a strong base (NaOH), the change in pH will be reflected in the change in EMF. When a small amount of alkali is added to the acid, a little change in the EMF is produced in the beginning. This change in electrode potential depends on the fraction of hydrogen ions removed. As an equivalence point is reached, the fraction of the hydrogen ions removed by constant volume of standard alkaliincreasesrapidly, thereby causing arapid changein theEMF.Thusif the EMFofthe cell is plotted against the volume of the standard alkali added, a curve is obtained. As the changeinEMFismuchmorerapidneartheequivalentpoint,theexactequivalentpointis

obtainedbydifferentialmethodwhere,agraphof$\frac{\Delta E}{\Delta V}$ Vs.volumeofalkaliadded,gives

themaximumofthecurvewhichcorrespondstoequivalencepointofthetitration.

$$HCl+NaOH \rightarrow NaCl+H_2O$$

Thecellcanberepresentedas

$$H_2(Pt)/acidsolution//KCl(aq)/calomelelectrode$$
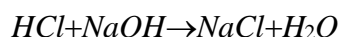
Procedure

EstimationofHCl:

10mLof0.1NHClispipettedoutinto100mLbeakerandsaturateditwithQuinhydroneand theindicatorelectrode(platinumelectrode)is   dipped.Theindicatorelectrodeandsaturated calomelelectrode(referenceelectrode)are connected tothepotentiometer.Thetwohalf cells are connected by means of a salt bridge. The potentiometer is standardized and used for measuringtheemfdirectly.1NNaOHistakenintheburetteandisaddedtotheHClsolution.

Theemftheacid,takeninthebeaker,ismeasuredinitially.Roughtitrationisfirstcarried outbyadding1mLofNaOHandtheemfforeachadditionismeasured.   Theemfdecreases graduallyandthenshowsasuddendecreaseintheemf.Enoughreadingsaretakenafterthe   sudden decrease in emf. From these rough titrations, the range of end point is determined.

Afterfindingtheendpointrange,fairtitrationsarecarriedoutbyrepeatedlyadding1mLis added and emf is measured for each addition and the readings are tabulated.

Graph is plotted with volume of alkali (NaOH) along X-axis and measured emf along Y-axis. A sigmoid curve is obtained and the equivalence point is noted at the end point of intersection.Toobtain asharpendpoint, anothergraphofvolumeofalkali(NaOH) along X-axis and$\frac{\Delta E}{\Delta V}$along Y-axis is plotted. The maxima obtained in the curve gives the accurate equivalence point.

Observations:

50.0 mL of 0.100 M HCl with 0.100 M NaOH
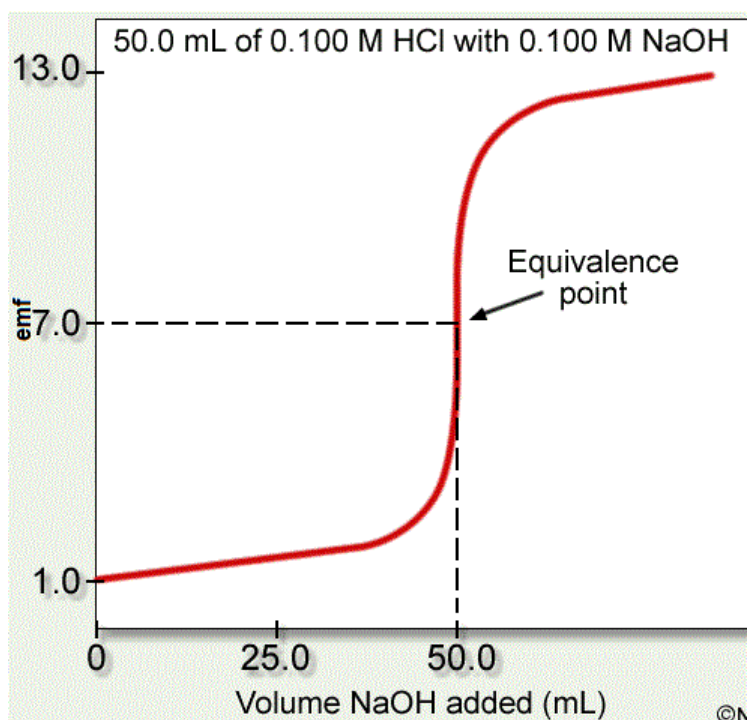
PilotTitration:

Volumeofalkali(NaOH)(mL)	EMF(mV)
0.0	
1.0	
2.0	
3.0	
4.0	
5.0	
6.0	
7.0	
8.0	
9.0	

FinalTitration

Volumeofalkali (mL)	EMF(mV)	$\Delta E/\Delta V$
0		
1.0		
2.0		
3.0		
4.0		
5.0		
6.0		
7.0		

8.0		
9.0		
10.0		
11.0		
12.0		
13.0		
14.0		
15.0		
16.0		

Calculations:

StrengthofHCl

*Volumeofacidtakeninbea*ker,V_1=20*mL*

Strengthofacid, N_1= ?

VolumeofNaOH $\left(fromthegraph\right)$,$V_2$= ....*mL*

StrengthofNaOH,N_2=.........................

AccordingtoVolumetricLaw

V_1N_1=V_2N_2

N_2=$\dfrac{V_1N_1N}{V_2}$

AmountofHCl

AmountofHClpresentinthegivensolution=…......................(N_2)×36.45g

RESULT:

1. TheequivalencepointofpotentiometrictitrationbetweenstrongacidVsstrongbaseis ... mL

2. TheNormalityofHClbytitratingwithNaOHusingpotentiometeris .... N.

3. TheamountofHClpresentinthegivensolution=…................g.

Experiment-6

EstimationofFe^{2+}byPotentiometryusingKMnO$_4$

Objective: Toestimate theFe^{2+}byPotentiometryusing KMnO$_4$.

Outcome : The student shall be able to analyze the variation of EMF values of given acid with addition of KMnO$_4$using Potentiometer.

Chemicals: KMnO$_4$, Distilled water, sulphuric acid, Ferrous sulphate solution.

Apparatus:volumetricflask,ptelectrode,saturatedcalomelelectrode,potentiometer

Principle:

Potentiometrictitrationisthetitrationinwhichpotentiometricmeasurementsarecarriedout in order to fix the end point. In this method, the interest is with the change in electrode potential, rather than with an accurate value for the electrode potential in a given solution. In a potentiometric titration, the change in cell e.m.f. occurs most rapidly in the neighbourhood of the end point.

> TheFe(II)–KMnO$_4$redoxsystemisrepresentedas

$$5Fe^{2+} + MnO_4^- + 8H^+ \rightarrow 5Fe^{3+} + Mn^{2+} + 4H_2O$$
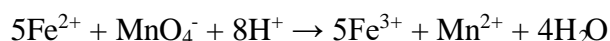
The determining factor is the ratio of the concentrations of the oxidised and the reduced forms of the iron species.

Forthereaction,

> Oxidisedform+ne-→Reducedform,

$$E = E^o + \frac{0.0591}{n} log \frac{[Ox]}{[Red]}$$

where E^0 is the standard Reduction Potential of the system. Thus the potential of the immersed electrode is controlled by the ration of these concentrations. During redox reactions, the potential changes more rapidly at the vicinity of the end point. The indicator electrodeisusuallyabrightplatinumwireorfoil,theoxidisingagentistakenintheburette. The cell can be represented as,

$$Calomel \parallel Fe^{3+}, Fe^{2+} \mid Pt.$$
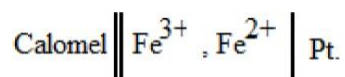
HerePtistheindicatorelectrodeandcalomelisthereferenceelectrode.

Procedure:

Preparationof0.1NKMnO$_4$:

0.1 N KMnO$_4$is prepared by dissolving 0.31 g of analar crystals in distilled water in a 100 ml volumetric flask. The solution is made up to the mark.

CalibrationofThePotentiometer:

Astandardcellofknownemfisconnectedtotheinstrumentanditsemfissetinthevoltage scale. The galvanometer key is pressed to complete the circuit and the deflection of the galvanometer needle is noted. If there is any deflection, the current passing through the rheostatisadjustedfornulldeflection.Thisproceduremakessurethatthevalueof emfwhich isreadonthescaleisthetruepotentialofthecellconsidered.Thepotentiometeriscalibrated using the Weston standard cell of potential 1.018 V.

EstimationOfFe (Ii):

- ThegivenFe(II)solutionis madeupto100mlinvolumetric flask.
- 20mlofthesolutionispipettedoutintoacleanbeaker.Tothis,25mlof2.5MH$_2$SO$_4$ and50mlofdistilledwaterareadded.

- A platinum electrode is dipped into this solution, and it is coupled with a calomel electrode through a salt bridge. The resulting cell is connected to the potentiometer.
- StandardKMnO4solutionisaddedfromtheburette,tothissolution,instepsof1mland the emf is recorded after each addition.
- At the end point, there is a jump in emf due to the absence of Fe^{2+}. The approximate range of the end point is determined.
- Theexperiment isrepeated by adding the titrant instepsof0.1 mlnear the endpoint. A graphisplottedbetweenemf,Eandthevolumeofdichromateadded.Theinflexionpoint gives the volume of titrant at the end point.
- The first derivative ($\Delta E/\Delta V$ vs. Volume of titrant) curves give the exact volume of dichromate required for the reaction. From the plot of E vs. Volume of titrant, potential at the equivalent point is obtained.
- AtomicweightofFeis 55.85
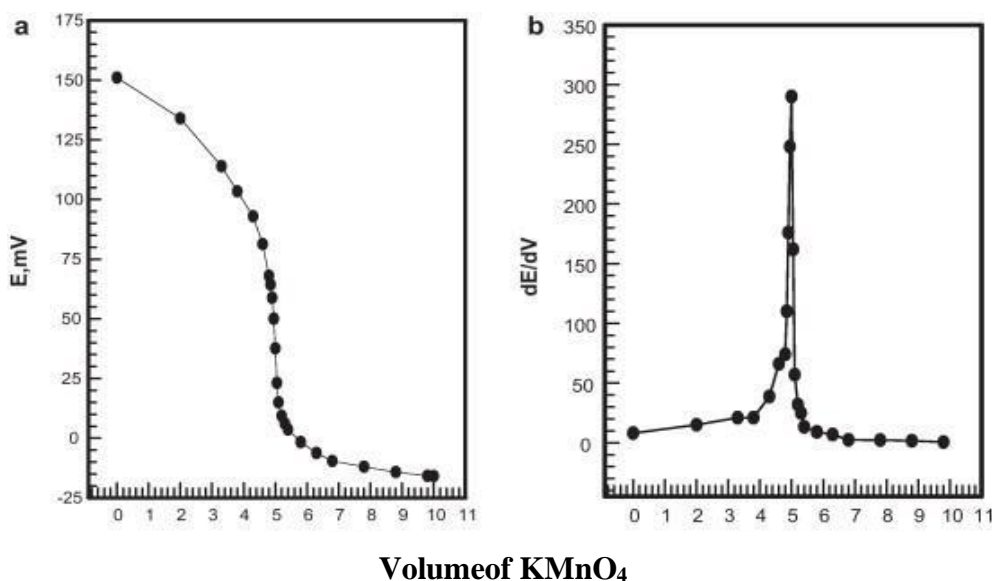
Volumeof KMnO4

Table:

S.No	Volumeof KMnO4	EMF	ΔE	$\Delta E/\Delta V$	V_{mean}
1					
2					
3					
-					
-					

Volume of $KMnO_4$ =

V_1ConcentrationofKMnO4=N_1=0.1N

Volume of Fe^{2+} solution = V_2= 20 ml

ConcentrationofFe2+solution=N_2=?

AccordingvolumetriclawN1V1= N2V2

$$N_2=N_1V_1/V_2$$

AmountofFe^{2+}ionpresentinthegivensolution=(N$_2$xEquivalentweightofFe^{2+})/10

$$=(N_2 \times 55.85/10) \text{ gm}$$

Result: TheamountofFe^{2+}ionpresentinthegivensolution=…................. gm

24

Experiment-7

DeterminationofRateConstantofAcidCatalyzedHydrolysisof Methyl Acetate

Objective: TodeterminetherateconstantofthehydrolysisofMethylacetateusing anacid as a catalyst.

**Outcome:**Thestudentshallbeabletoidentifyandanalyzetherateconstantvaluesof methyl acetate hydrolysis at regular intervals of time.

Chemicals: Methyl acetate, distilled water, HCl, NaOH, Phenolphthalein indicator, 0.1 N oxalic acid.

Apparatus:Conicalflask,volumetricflask,burette,pipette,reagentbottles.

Principle:

Methylacetateundergoes hydrolysis,inthepresenceofanacid(HCl,for example),togive acetic acid and methyl alcohol.

$$CH_3COOCH_3 + H_2O \rightarrow CH_3COOH + CH_3OH$$
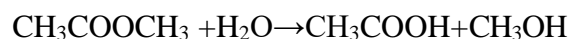
In the presence of an acid, this reaction should be of second order, since two molecules are reacting.But, itisfound tobefirst order.Thismaybeexplainedin thefollowingway :The rate of the reaction is given by

$$dx/dt = k^1[CH3COOCH_3][H_2O]$$

wherek'istherateconstant(orspecificrateconstant).Sincewaterispresentinlargeexcess, its active mass (molar concentration) virtually remains constant during the course of the reaction. Therefore, its active mass gets included in the constant, and the above equation reduces to:

$$dx/dt = k^1[CH_3COOCH_3]$$

Thus, the rate of the reaction is determined by one concentration term only (that is, by a single power of the concentration term only). Hence, the reaction is first order. Such reactions are also referred to as pseudo first order reactions. The progress of the reaction (hydrolysis of ester) is followed by removing a definite volume of the reaction mixture, at definiteintervalsoftime,coolingitinice,andtitratingtheaceticacidformedagainstalkali, which has already been standardized. The amount of alkali used is equivalent to the total amount of hydrochloric acid present originally and the amount of acetic acid formed in the reaction.

The amount of acetic acid formed (x), at definite intervals of time (t), can be obtained. The amount of acetic acid formed, at the end of the reaction, is equivalent to the initial concentration (a) of the ester. Suppose the volumes of the sodium hydroxide solution (titre value) required for neutralization of 5 ml of the reaction mixture are :

(i) atthecommencementofthereactionis V_o

(ii) aftertime(t)is V_t

(iii) attheendofthereactionis V_∞The

n:

x(amountofaceticacidformed after time)isproportionalto($V_t - V_o$). a

(initial concentration of ester) is proportional to ($V_\infty - V_o$).

[a–x](concentrationofesterpresentaftertimet) isproportionalto (V∞ -

Vo) - (Vt -Vo) = (V∞ -Vt)

Thefirstorderrateexpressiongivenby:

$$k_1 = \frac{2.303}{t} \log \frac{a}{[a-x]} \quad \text{would correspond to :}$$

$$k_1 = \frac{2.303}{t} \log \frac{(V_\infty - V_o)}{(V_\infty - V_t)}$$

Hence,therateconstant(k_1) couldbe calculated.

Procedure

Step I:

StandardizationofNaOHusingstandardOxalicacid(0.1N)

1. Pipetteout10mlofgiven0.1NstandardOxalicacidintoa100mlconicalflask.

2. TitratethissolutionagainstthegivenunknownconcentrationofNaOHusingphenolphthalein indicator until the end point is colorless to pale pink.

3. Tabulatethevaluesandrepeatthetitrationforconcurrentreadingsanddeterminethe unknown concentration of supplied NaOH solution.

Table 1

S.No	Volume of Oxalic Acid taken (ml)	Burette Readings (ml) Initial	Final	Volume of NaOH consumed (ml)
1				
2				
3				

$$N_1V_1 = N_2V_2$$

Here,N1=concentrationofoxalicacid,V_1=Volumeofoxalicacid N_2=

concentration of NaOH, V_2= Volume of NaOH

Step II:

Standardization ofHClusingNaOH solution

1. Pipetteout2mlof givenHClinto a100mlconicalflask.

2. TitratethissolutionagainsttheNaOHusingphenolphthaleinindicatoruntiltheendpoint is colorless to pale pink.

3. Tabulatethevaluesandrepeatthetitrationforconcurrentreadingsanddeterminethe unknown concentration of supplied HCl solution.

Table 2

S.No	Volume of HCl taken (ml)	Burette Readings (ml) Initial	Final	Volume of NaOH consumed (ml)
1				
2				
3				

$(NaOH) N_2 V_2 = N_3 V_3 (HCl)$

Concentration of HCl, $N_3 =$ _____

Step III:

Determinationofrateconstant(k1)fortheacid-catalyzedhydrolysisofmethylacetate

1. Take100mlofgivenHCl(whosestrengthisdeterminedinstepII)solutioninastoppered reagent bottle.

2. Add 5 ml of methyl acetate solution to the HCl solution. Note the time when half of the methyl acetate solution is added. The mixture is shaken well.

3. Pipetteout5 mlof thereactionmixture anddischargeit into50 mlof ice coldwaterkept in a conical flask.

4. Titrate the reaction mixture against NaOH solution using phenolphthalein as indicator. This titre value corresponds to Vo.

5. Repeatsteps3and4atintervalsof5,10,15,20,30,45,60minutes.Eachtitrevalue corresponds to Vt.

6. Taketheremaining solution in astoppered conicalflask and heated to 60°C, and keep at this temperature for 5 minutes.

7. Thesolutionisallowedtocool toroomtemperature.

Table 3

S.No	Time (min)	Volume of solution taken (ml)	Burette Readings (ml) Initial	Final	Volume of NaOH consumed (ml)	$(V_\infty - V_t)$ (ml)	$\log(V_\infty - V_t)$	$k_1 = \dfrac{2.303}{t} \log \dfrac{(V_\infty - V_0)}{(V_\infty - V_t)}$ (min^{-1})
1	0 (V_0)							
2	5 (V_{t1})							
3	10 (V_{t2})							
4	15 (V_{t3})							
5	20 (V_{t4})							
6	30 (V_{t5})							
7	45 (V_{t6})							
8	60 (V_{t7})							
9	V_∞							

8. RepeatSteps3and4.ThistitrevaluecorrespondstoV$_\infty$tillconcurrentvaluesareobtained.

9. Plotagraphof log(V$_\infty$ -Vt)versustime(t) anddeterminethe slope.

10. Reportthetheoreticalandgraphicalvalueofrateconstant(k$_1$).

ObservationsandCalculations:

RoomTemperature=………......^{o}C;V$_\infty$=……..ml;V$_o$=……..ml;(V$_\infty$-Vo)=...........................ml ;

log(V$_\infty$-Vo) =..............

Mean Value of Rate Constant (k1) = _____

Results:

1. Strength of NaOH Solution = _____

2. Strength of HCl Solution = _____

3. RateConstant(k$_1$)fortheacid-catalyzedhydrolysisofmethylacetate   at…^{o}C= =_____(theoretical).

= _____(graphical).

Experiment-8

SynthesisofAspirinandParacetamol

Objective: Tosynthesizesomecommonpainrelievers:aspirinandParacetamol.

Outcome : The student shall be able to synthesize the crude sample of Aspirin and Paracetamol.

Principle: Aspirin (acetylsalicylic acid) is both an organic ester and an organic acid. It is used extensively in medicine as a pain killer (analgesic) and as a fever-reducing drug (antipyretic).Wheningested,acetylsalicylicacidremainsintactintheacidicstomach,butin thebasicmediumoftheupperintestinaltract,ithydrolyzesformingthesalicylateandacetate ions.

Aspirin(molarmassof180.2g/mol)ispreparedbyreactingsalicylicacid(138.1g/mol)with acetic anhydride (molar mass of 102.1 g/mol). Aspirin is a weak monoprotic acid.

salicylic acid   acetic anhydride   aspirin (acetylsalicylic acid)   acetic acid
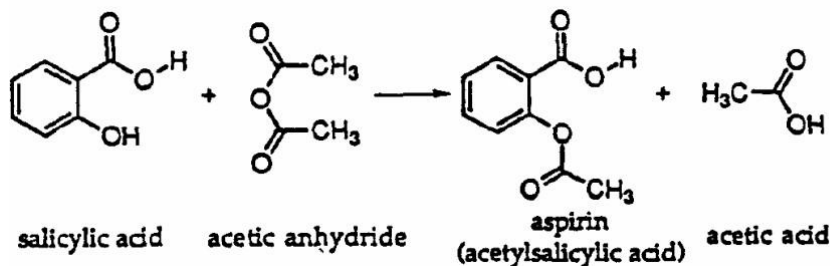
Paracetamol(acetaminophen)actsasafeverreducerandpainreliever.Acetaminophenisan amide, a compound that is a derivative of ammonia that has been reacted with an acidic substance,inthiscase,aceticacid.Itcanbefoundinseveralanalgesicpreparations,suchas Tylenol, some of which may contain other ingredients such as caffeine and buffers.

p-Aminophenol   Acetic anhydride   Acetaminophen   Acetic acid
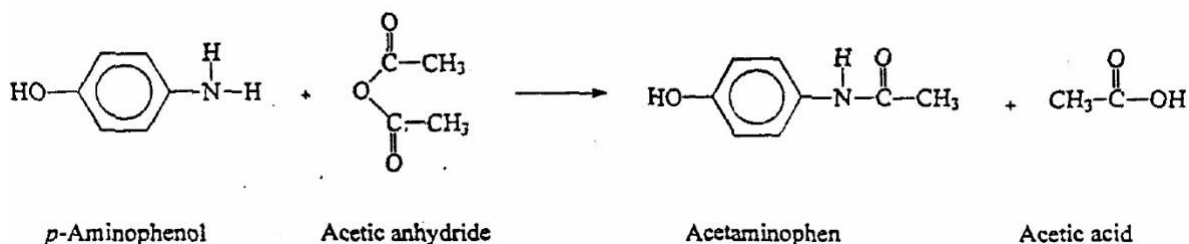
PROCEDURE

a) PreparationofAspirin

Chemicals: Salicylic acid,aceticanhydride,sulfuricacid,ethanol,ice.

Apparatus: Dropper,Erlenmeyerflask125mL,Beakers,Graduatedcylinders,Watchglass, Stirringrod,Ringstand,Buchnerfunnel,filterpapertofitBuchnerfunnel,vacuumfiltration flask, Rubber tubing for vacuum flask, thermometer, dropper.

Procedure

- Weighout2.0gofsalicylicacid.Placeitin a125mLconicalflask.Add5mLofacetic anhydride.
- Swirl the flask to wet the salicylic acid crystals. Add 5 drops of concentrated sulfuricacid, to the mixture.
- Gentlyheattheflaskinaboilingwaterbathforabout10 minutes.
- Removetheflaskfromthehotwaterbathandadd10mlofdeionizedicewaterto decompose any excess acetic anhydride.
- Chillthesolutioninanicebathuntilcrystalsofaspirinnolongerform,stirringoccasionally to decompose residual acetic anhydride.

- If oil appears instead of a solid, reheat the flask in the hot water bath until the oil disappears and again cool.
- Setupavacuumfiltrationapparatus.WetthefilterpaperintheBuchnerfunnelwith1-2 ml of distilled water.
- Turn on the water aspirator. Decant the liquid onto the filter paper, minimizing any transfer of the solid aspirin.
- Add15mlofcoldwatertotheflask,swirl,andchillagain.Pourtheliquidandthecrystals of aspirin onto the filter paper. Repeat until the transfer of the crystals to the vacuum filter is complete.
- Determinethe massofthecrude aspirin crystals.

b) Preparation of Paracetamol (Acetaminophene)

Chemicals: Aceticanhydride,Phosphoricacid,Ethanol

Apparatus: DropperErlenmeyerflask,125mLBeakers,Graduated cylinders,Watchglass Stirring rod,, Vialto hold aspirin sample, Ring stand Clamp (to hold 125-mLconicalflask) Buchner funnel, Filter paper to fitBuchner funnel,Vacuumfiltration flask, Rubber tubing, Ice, Dropper.

Procedure:

- Filla400mLbeakerabouthalffullwithwater.Placethebeakerandwateronahotplate and bring to a boil.
- Weigh out 1.5 g of p-aminophenol and transfer it into a 125 mL conical flask. (Avoid contact with skin. You may wish to wear gloves.)
- Add 25 mL of water. Add 20 drops of concentrated phosphoric acid (H_3PO_4), and swirl theflaskuntilalloftheaminedissolves.Ifnot,adda fewmoredropsofphosphoricacid.
- Turn off the hot plate. Place the flask in the hot water. Carefully add 2 mL of acetic anhydride to the flask. Leave the flask in the warm water for 10 minutes.
- Remove the flask and place it an ice-water bath. Stir the mixture to crystallize the acetaminophen.Youmayneedtoscratchthewallsoftheflasktostartthecrystallization. If no crystals appear, add a small seed of acetaminophen to start the crystal formation. Allow the flask to stay in the ice-water bath for 30 minutes.
- Collectthecrystals inaBuchnerfunnelusingvacuumfiltration.Washthecrystals with 10 mL of cold water. Allow the crystals to dry.
- Determinethemassofthecrudeacetaminophen.

Results:

Massofsalicylicacid=.................... gms

Massofcrudeaspirin=.....................gms

Yieldofaspirin=............ %

MassofP-aminophenol=.....................gms

Massofparacetamol=....................gms

Yieldofparacetamol =................ %

Experiment-9

ThinLayerChromatographyCalculationofRfValues.Eg
Ortho and Para Nitro Phenols

Objective: To calculationtheRfValues oforthoandparanitrophenols byusingthinlayer chromatography.

Outcome: ThestudentshallbeabletoidentifyandcalculatetheRfvaluesofvarious compounds present in the given mixture.

Chemicals:O-nitrophenol,P-nitrophenol,ethylacetate,di-chloromethane

Apparatus:TLCplate,capillary tubes,

Principle:

Mostreactionsproducemorethanoneproduct.Naturallyoccurringmaterialsareonlyrarely 100%pure.Itisthereforedesirabletohaveasimple, fastandefficientwaytodeterminethe purity of Organic mixtures. The separation of a mixture by passing it, in solution, over an adsorbent (such as Alumina or Silica Gel) is the basic idea of <u>Chromatography.</u> It involves the passage of a mobile phase across a<u>stationary phase</u>in a column. Usually a mixture of compoundsispresentinthe<u>mobilephase.</u>Assoonasthemixturecomesincontactwiththe stationary phase, some or all of the components of the mixture are adsorbed on it. As additional mobile phase comes along, some or all of the mixture will dissolve and continue moving. This adsorption/solution process continues along the length of the column. If a properchoiceofmobilephase,stationaryphase,solventandotheroperatingparameterswas made, the mixture will be separated in the column and its various components will emerge at different times.

In Thin Layer Chromatography, a liquid solution is directly applied to a solid adsorbent. CapillaryactiondrawsadevelopingsolventuptheTLCplate.Asthissolventpassesthrough the spot, the mixture will be dissolved and will begin to move with the solvent front. However,theadsorbentwillalsoreabsorbpartorallofthemixture.Asmoresolventcomes by, the mixture will again go into solution, move further and be reabsorbed. Since different materialswillbedissolvedandreabsorbedatdifferentrates,separationwilltakeplace.This passage of the solvent front through the adsorbent is known as **developing**the plate. The extent of separation, measured by retention factor ("R$_f$") value differences, will depend on therelativesolubilitiesandrelativestrengthsofadsorptionofthecomponentsofthemixture. Organiccompoundsinteractwithabsorbentsbyavarietyofinteractions.Ifthecompoundis non-polar, it can only have weak 'Van der Waals' attractions for the absorbent. However, more polar molecules may interact more strongly by a variety of mechanisms including dipole-dipoleinteractions,coordination, and hydrogenbonding. Themostimportantruleof chromatography is that *the more polar compounds will be absorbed most strongly on absorbents (stationary phases), while non-polar compounds will be only very weakly absorbed.*Inatypicalchromatographyexperiment,thenon-polarcompounds,sincetheyare poorly absorbed, will be held least strongly and will move quickly through the plate. Polar compounds, on the other hand, will be slowed on their process through the plate by their stronginteractionswiththesolid phase. This separationbasedonpolarity willexplain most of the chromatography encountered in this course.

TypesofAdsorbents usedinChromatography

Listedindecreasingpowerof adsorption:

Alumina>ActivatedCharcoal>MagnesiumSilicate>Silica>Starch

Solvents CommonlyUsed inChromatography

Listedindecreasingpolarity:

AceticAcid>Water>Methanol>Acetone>Ehtyl acetate>Diethylether>Chloroform> Methylene chloride > Toluene > Cyclohexane > Petroleum ether

Foratypicalseparation,avarietyofdifferentcombinationsofsolventandadsorbentmaybe effective.Onceyouhavedevelopedyourplate,itmustbe **visualized**.Thisvisualizationmay be accomplished by reacting the developed plate with a chemical reagent. Iodine (I_2) is one oftheeasiesttouseoftheseveralcommonchemicalvisualizingagents.Thedevelopedslide is simply exposed to I_2vapors in a chamber similar to the developing chamber for a few minutes.AlmostallcompoundswillformaweakcoloredcomplexwiththeI_2.Thiscomplex will appear as a darker area on the slide. The 'spots' are characterized by their$R_f$value,a measure of how far the spot traveled with that combination of adsorbent and solvent.

Procedure:

- Take 1 TLC plate handle it only on the edges, as fingerprints contain UV-active materials.
- Usingapencildrawaverylightlineacrossthesheet(shortdimension)about1cmfrom one end.
- Then make4 smalllightmarks ateven intervals along thelinefor spotting thesamples. Draw another light line about 1 cm from another end of the plate for the solvent front.
- Obtain a TLC chamber and place solvent, a 5% ethyl acetate in dichloromethane to 0.5 cm height.
- Placeapieceoffilterpaperaroundtheinsidesurfaceofthecontainerandextendintothe solvent.
- AglassjarwithalidorabeakerwithawatchglassoracoverofaPetridishcanbeused as a TLC chamber:
- Using clean capillary tubes carefully spot four samples at two pencil marks as shown below.
- The spots should be as small as possible in order to minimize tailing and overlapping when the TLC plate is developed. If a more intense spot is desired, let the spot dry and re-spot in the same location.
- When the spots are dry, place the TLC plate in the developing chamber. Then gently close the chamber.
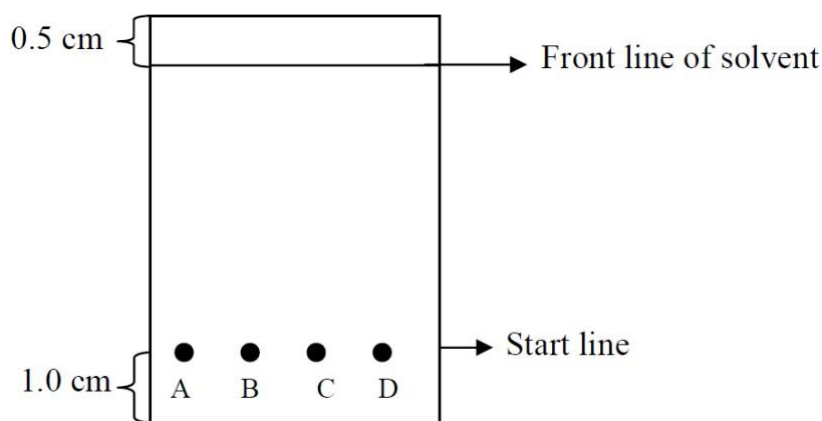
A:o-Nitrophenol   B:p-Nitrophenol

- Be sure that the bottom edge of the TLC plate is in the solvent but the spots are above the solvent, and the filter paper does not touch the chromatographic sheet.
- Whenthesolventhasmovedtothefrontline,removetheplate.Layitonacleansurface in well ventilated area and allow the solvent to evaporate until the plate appears dry.
- VisualizetheplateunderiodinechamberMeasureallthedistancestraveledbythe compounds and solvent.

- Calculatetheretentionfactor(R*f*)foreachcompound.andimmediatelydrawalight pencil line around each spot.

Calculationofretentionfactor:
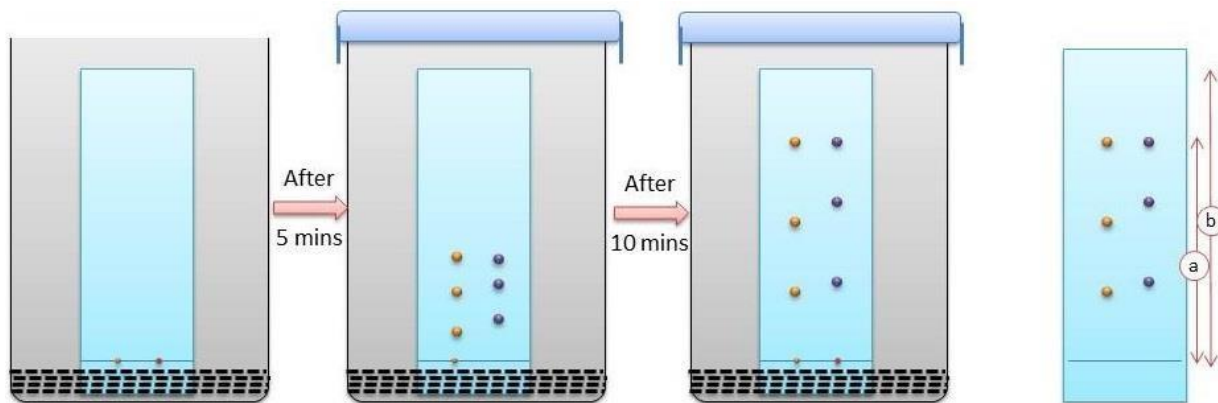
$$Rf = \frac{\text{distance travelled by the component}}{\text{distance travelled by the solvent}}$$

$$= \frac{a}{b}$$

Retention factor Rf $= \dfrac{\text{Distance travelled by the component}}{\text{Distance travelled by the solvent}}$

Result:

Retentionfactorofo-nitrophenol=…………….

Retention factor of p-nitro phenol = ……………

Experiment-10

DeterminationofAcidValueofCoconutOil

Objective: ToDeterminetheAcidValueofCoconutOil.

Outcome: Thestudentshallbeabletoidentifyandcalculatetheacidvalues ofcoconutoil.

Chemicals: NaOH,distilledwater,Phenolphthaleinindicator,potassiumhydrogenphthalate, coconut oil, ethanol, diethyl ether

Apparatus: Volumetricflask,conicalflaskpipette**,**burette,weighingbalance, beaker

Principle:

Acidity may occur in coconut oil upon storage due to the decomposition of the oil to free fatty acids, peroxides and low molecular weight aldehydes and ketones, which produce distinctive smell and affect the quality of oil. Acid value may be defined as the number of milligrams of KOH required to neutralise the free acid in 1gm of fatty oil.

Procedure:

PreparationofSolutions:

1) **0.1NSodiumhydroxide:** Weighaccurately0.4gmofsodiumhydroxideinto100mL standard flask, dissolvein littledistilled water and makeup thesolution to themark with distilled water and shake the flask well for uniform concentration.

2) **Ethanol-ethersolution:** Prepareamixtureofethanolanddiethylether(1:1v/v);add 1mL of phenolphthalein indicator and titrate with NaOH till pale pink colour is observed. This is the neutralisation of ethanol-ether solution.

3) **Standardsolutionofpotassiumhydrogenphthalate:** Accuratelyweighabout 1.2gm of potassium hydrogen phthalate into 100mL flask dissolve in little water make up the solution to the mark with distilled water.

Procedure:

1) **Standardisation of NaOH solution:** Pipette out 20mL of potassium hydrogen phthalate solution into a 250mL conical flask and add 2 drops of phenolphthalein indicatorand titratethesolutionwith NaOHtaken in burette.Palepink colour isthe end point of the titration. Note the burette reading and let the value be x mL

2) **Standardisationofcoconutoil:** Accuratelyweigh1gmofcoconutoilintoa250mL conical flask; add 50mL of ethanol-ether solution. Shake the flask well for uniform dissolution of the sample. Add 1 drop of phenolphthalein indicator and titrate the solution with NaOH taken in the burette till pale pink colour is observed. Note the burette reading, let the value be y mL

Calculations:

(M_1) Molarityofpotassiumhydrogenphthalate $= \dfrac{1.2 \times 1000}{204.22 \times 100} = 0.058M$

(M_2) Molarity of NaOH: $\qquad M_1V_1/M_2V_2$

$$M_2 = M_1V_1/x$$

Acidvalueofcoconutoil $= \dfrac{M_{NaOH}}{W}$ (W=wightofcoconutoil=1 gm)

Result: Theacidvalueofgivenoilis …………..

Experiment-11

VerificationofFreundlichAdsorptionIsotherm-Adsorption of

AceticAcidonCharcoal

Objective: To study the adsorption of acetic acid from aqueous solution by activated charcoal & examine the validity of Freundlich & Langmuir's isotherm.

Outcome : The student shall be able to identify and calculate the adsorption values & plot the adsorption isotherms.

Chemicals: 0.1N acetic acid solution, 0.1N sodium hydroxide solution, phenolphthalein indicator, powdered activated charcoal etc.

Apparatus: Fivereagentbottleswithstoppers,burette,pipette,conicalflasks

Principle: Adsorption is accumulation of a substance at an interface. The adsorption of a solutefromasolution,generallyfollowstheFreundlichempiricaladsorptionisothermgiven by

$$\frac{x}{m} = KC^{\frac{1}{n}}$$

Where

x = weight of adsorbent, m = mass of adsorbent, K = constant representing the capacity of the adsorbent, and C = equilibrium concentration of the solution.

From the concentration (C), the volume of each original solution and concentration of each solutionafter equilibrium (Ce), the weight ofoxalic acidpergramof the adsorbent (x/m) is calculated as follows.

$$\frac{x}{m} = \frac{(C - C_e) \, X \, 60}{10}$$

Drawagraphoflog(x/m)vs.logCeandcalculatetheconstantnandK usingtheequation.

$$\log \frac{x}{m} = \log K + \frac{1}{n} \log C_e$$

Procedure:

1. Takefiveclean,drystopperreagentbottles&labelthemfrom1to 5.
2. Add by means of a burette 50, 40,30, 20 &10 ml of 0.1N acetic acid solution & 0, 10, 20, 30& 40 ml distilled water in bottle nos. 1,2,3,4 & 5 resp.
3. Weigh accurately 1 gm of activated charcoal & add to bottle no.1. Similarly, add 1 gm activated charcoal in each of the remaining bottles. Stopper the bottles & shake them well.
4. Meanwhile determine the initial concentration of acid by titrating against std. NaoH solution using phenolphthalein indicator.
5. Usingdryfilterpapersofdualsizefilterthesolutionofeachbottleinseparatedryflasks. Titrate 10 ml of filtrate from each bottle with 0.1Nn NaOH using phenolphthalein indicator.Stakethreereadingsforeachbottle&takethemean.Thesereadingsgivesthe equilibrium concentrations of the solution i.e. concentration of acid after adsorption. Tabulate the result.

Observationtable:

Bottle	Initial	Equilibrium	Amountof	x/m	logx/m	logCe

No.	conc. of acidC_0m eq./lit	Conc.ofacid, Ce gm eq./lit	acid adsorbed (x)			
1						
2						
3						
4						
5						

Here $x = \dfrac{(C0 - Ce) \times V \times Eq.wt.of\ the\ acid}{1000}$

Where,V =totalvolume(50ml)ofthesolutionineachbottle.

Calculation:

Calculate initial concentration (C_0) of acetic acid in gm equivalent per lit for each sample. Also calculate the equilibrium concentration (Ce) of theacid solution in each of thebottles in gm equivalent per lit. Then calculate the amount of the acid adsorbed in each bottle as follows:

Graph:

1. Plotthegraphoflog(x/m)(Y-axis)againstlogCe(X-axis).Astraightlinewillbeobtained This is

in agreement with Freundlich equation. Find slope & Y-intercept values.

2)PlotCe/(x/m)(Y-axis)againstCe(X-axis).Astraightlineobtainedshowstheagreement with Langmuir adsorption isotherm. Calculate slope & Y-intercept.

Result:

ThusFreundlich& Langmuiradsorptionisotherm arestudiedbytheaboveExperiment.

Experiment-12

DeterminationofViscosityofCastor OilandGroundNutOilby

UsingOstwald's Viscometer

Objective: Todeterminetheviscosityofthegivencastoroilandgroundnutoil.

Outcome: Thestudentshallbeabletodetermineandcalculatetheviscositycoefficientvalues of Castor oil and groundnut oil.

Chemicals:astoroil andgroundnutoil,distilled water.

Apparatus: Specific gravity bottle, viscometer, rubber tube with screw pinch cock, stand, beaker

Theory: Theforceoffrictionwhichonepartoftheliquidofferstoanotherpartoftheliquid is called viscosity. For measuring the viscosity coefficient, Ostwald viscometer method is used which is based on Poiseuille's law. According to this law, the rate of flow of liquid through a capillary tube having viscosity coefficient 'η' can be expressed as

$$\eta = \frac{\pi \cdot r^4 tP}{8vl}$$

where,v=vol.ofliquid(inml)

t=flowtime(insec.)throughcapillary r=

radius of the capillary (in cm)

l=lengthofthecapillary(incm)

P=hydrostaticpressure(indyne/sq.cm)

η= viscosity coefficient (in poise).

Since, the hydrostatic pressure (the driving force) of the liquid is given by P = dg h (where h is the height of the column and d is the density of the liquid);

ηαPt;or, ηαdght

If,$\eta_1$and$\eta_2$aretheviscositycoefficientsoftheliquidsunderstudy,d_1,$d_2$aretheirdensities and t_1 and t_2 are their times of flow of *equal volume* of liquids through the same capillary respectively, then

η₁αd₁ght₁

$\eta_1 \alpha d_1 ght_1$

$\eta_2 \alpha d_2 ght_2$

Hence

$$\frac{\eta_1}{\eta_2} = \frac{d_1\ t_1}{d_2\ t_2}$$

Here,usuallytheviscosityofgivenliquidismeasuredwithrespecttowaterwhoseviscosity is known very accurately at different temperatures. The SI physical unit of viscosity is the pascal-second(**Pa·s**),(i.e.,kg·m−1·s−1).Thismeans:ifafluidwith aviscosityofone **Pa·s** is placed between two plates, and one plate is pushed sideways with a shear stress of one pascal, it moves a distance equal to the thickness of the layer between the plates in one second. Thecgs unitfor thesameis the **poise** (P),(named after J. L. Marie Poiseuille). Itis more commonly expressed, as **centipoise** (**cP**). [1 cP = 0.001 Pa·s]. Water at 20 °C has a viscosity of 1.0020 cP.

Procedure:

1. Notethelaboratorytemperature.

2. Washthedensitybottlewithdistilledwateranddry.

3. Take the weight of the empty & filled (with distilled water) specific gravity bottle (with stopper).Then,weighthefilledwithspecificgravitybottleunknowngivenliquids

individually.Usethedataformeasuringthedensities.

4. Cleanandrinsetheviscometerproperlywithdistilledwater.Fixtheviscometervertically onthestandandfilledwithspecificamount(say20ml)ofmixture(everytimetakethesame volume).

5. Timeofflows wasrecordedforeachsolution(waterandthegivenliquids).

6. Take3to4readings.

Observations:

1. Laboratorytemperature=….^{0}C

2. Densitymeasurement:

Weightofemptydensitybottle(w1)=… .... g.

Weightofdensitybottlewithwater(w2)=…............. g.

Weightofdensitybottlewithcastoroil(w3)=…............ g.

Weightofdensitybottlewithcastoroil(w4)=…............ g.

So,

Weight of water = (w2-w1) =…g.

Weightofcastoroil=(w3-w1)=…g.

Weightof ground nutoil= (w4-w1) =…g

Sample	Flowtimes			
	t_1	t_2	t_3	Average
Water				
Castor oil				
Ground ut oil				

Calculations:

Determinationoftheviscosityofthe liquid(η)

As discussedwarlier

$$\frac{\eta_c}{\eta_w} = \frac{d_c\ t_c}{d_w\ t_w}$$

$$\eta_c = \frac{d_c\ t_c}{d_w\ t_w} \text{X}\ \eta_w$$

here η_c=viscosityof castoroil;η_w=viscosityofwater(1.0020cP at20^{0}C) d_c =

density of castor oil; dw = density of water

t_c=flow timeforcastoroil;t_w=flowtimeforwater

$$\frac{\eta_g}{\eta_w} = \frac{d_g\ t_g}{d_w\ t_w}$$

$$\eta_g = \frac{d_g\ t_g}{d_w\ t_w} \text{X}\ \eta_w$$

here η_c=viscosityofgroundnut oil; η_w=viscosityofwater

d_c = density of ground nut oil; dw = density of water

t_c=flowtimeforgroundnut;t_w=flowtimeforwater

Result:

Theviscosityof thegivencastoroilwithrespectto wateratlaboratorytemperaturewas found to be.......................... CP.

Theviscosityof thegivengroundnutoilwithrespect towateratlaboratorytemperature was found to be..................... CP.

Experiment-13

DeterminationofPartitionCoefficientofAceticAcidbetween n-Butanol and Water

Objective: To determine the partition coefficient of acetic acid between n-butanol and water.

Outcome: The student shall be able to determine and calculate the Partition coefficient values of n-butanol and water.

Chemicals: Acetic acid, n-butanol, distilled water

Apparatus: Stoppard bottle, beaker, pipette, conical flask

Principle:

In dilute solutions at constant temperature a solute which exists in the same molecular species in two non-miscible solvents, will distribute itself between these two solvents at constant temperature according to the partition law, the partition coefficient:

$k = C_1/C_2$

where

C_1 = concentration of acetic acid in water, and

C_2 = concentration of acetic acid in butyl alcohol

The concentrations C_1 and C_2 must be expressed in the same units either as grams, gram molecules, or gram equivalents per litre.

Procedure:

- Boil about 200 mL of distilled water in a beaker for 10 minutes. Pour into a flask and stopper lightly and cool. This is CO_2 free water for later use.
- In a 200 mL glass stoppered bottle place about 70 mL of approximately 2M acetic acid and 50 mL of n-butyl alcohol.
- Stopper the bottle and shake well for at least 1 minute, and then allow the liquid layers to separate. Note and record the temperature of the mixture.
- Insert a 25 mL pipette and carefully withdraw a 25 mL aliquot of the upper alcohol layer. The pipette should first be rinsed by sucking up a little of the solution and discarding this.
- Pipette the solution into a second glass stoppered bottle and add an approximately equal volume of boiled distilled water to this second bottle.
- Shake-well to transfer the acid to the water layer, add 3 drops of phenolphthalein and titrate with the approximately 1.0 M sodium hydroxide. The bottle should be stoppered from time to time and vigorously shaken and titration continued until a faint permanent pink colour remains.
- Pipette, also a 25 mL aliquot from the lower aqueous layer of the first bottle as follows. Close the pipette with the finger and place it carefully in the lower layer.
- Suck up and blow out gently as small quantity of liquid to wash out any small quantity of the upper layer that has got into the pipette. Rinse the pipette with a little of solution.
- Allow the solutions to settle and withdraw 25 mL of the lower solution. Place this solution in a flask, add three drops of phenolphthalein and titrate with the approximately 1.0 M sodium hydroxide.
- Add about 25 mL each of fresh butanol and boil distilled water (but no further acid) to the original mixture remaining in the first bottle. Repeat the procedure above and sample and titrate with the new concentrations of acid.

The following table summarises the above procedure:

Initial mixture: 50 mL n-butyl alcohol, 70 mL 2M acid:

Summaryof procedure

AlcoholLayer		Water Layer		Alkalifor TitrationmL		K= C1/C2
Sample Removed ml	Fresh Alcohol Added	Sample Removed Ml	Water Added ml	Water Alcohol C1	C2	
25	--	25	--			
--	25	--	25	--	--	
25	--	25	--			

Result: Thepartition coefficientofaceticacidbetweenn-Butanolandwateris…………

Experiemnt-14

DeterminationofSurfaceTensionofaGivenLiquidUsing Stalagmometer

Objective: Determinethesurfacetensionofagivenliquidatroomtempusingstalagmometer by drop number method.

Outcome : The student shall be able to determine and calculate the surface tension values of reference liquidand given liquid.

Apparatus: Stalagmometer,specificgravitybottle,asmallrubbertube,screw pinchcork

Chemicals: Distilledwater,experimentalliqui

Principle: Inthedropnumbermethod,thenumberofdropsformedbyequalvolumesoftwo liquidiscounted.If$m_1$and$m_2$isthemassofonedropofeachoftheliquidhavingdensities d_1 and d_2 respectively. If n_1 and n_2 is the number of drops formed by volume v of the two liquids, then their surface tensions are related as

$$¥_1/¥_2= (d_1/d_2)*(n_2/n_1)$$

Oneoftheliquidiswateritssurfacetensionanddensityareknown.Thenthesurfacetension of the given liquid can be calculated.

Procedure:

1. Cleanthestalagmometerwithchromicacidmix,washwithwateranddryit

2. Attach a small piece of rubber tube having a screw pinch cock at the upper end of the stalgmometer.

3. Immerse the lower end of the stalagmometer in distilled water and suck the water 1-2cm above mark A. adjust the pinch cork so that 10-15 drops fall per minute .

4. Clamp the stalgmometer allow the water drops to fall and start counting the number of drops when the meniscus crosses the upper mark A and stop counting when the meniscus passes mark B

5. Repeat theexercisetotake three tofour readings

6. Rinsethestalgmometerwithalcoholanddryit

7. Suckthegivenliquidinthestalgmometerandcountthedropsas incaseofwater

8. Takeacleandryweighingbottleweighsitwithwateraswellaswithliquid.

9. Notethetempofwatertakeninabeaker.

Observations:

Room temp = t^0C

Densityofwater=d_w

Surfacetensionofwater=¥dynes/cm

	Noofdrops FromaFixedVolume			Mean
Liquid	1….	2……	3…..	n_l=
Water	1….	2…….	3……	n_w=

Weight of empty specific gravity bottle = $w_1$gram

Weight of specific gravity bottle + water = w_2 gram

Weightofemptysp.gravitybottle+liquid=$w_3$gram

Weight of water= (w_2-w_1) gram

Weightofliquid=(w_3-w_1)gram

Calculations:

Densityoftheliquid

$D_l = (w_3-w_1)/(w_2 - w_1) * d_w$

Surfacetensionof liquid=

$¥_l=(d_l/d_w)*(n_w/n_l)*¥_w$

Result

Thesurfacetensionofliquidis………dynes/cm.

Densityoftheliquid

$D_l = (w_3-w_1)/(w_2 - w_1) * d_w$

Surfacetensionof liquid=

$¥_l=(d_l/d_w)*(n_w/n_l)*¥_w$

ADDITIONAL

EXPERIMENTS

EXPERIMENTNO-15

ESTIMATIONOFMOHR'SSALT USINGSTANDARDKMnO$_4$SOLUTION

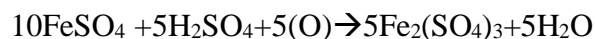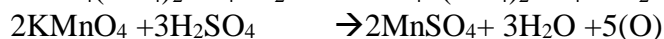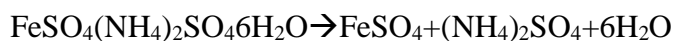**Objective:** Toestimatetheamountof Ferrousammoniumsulphatepresentinonelitresolution using 0.02M KMnO$_4$ solution.

Outcome:Thestudentshallbeableto identifytheoxidationof Ferrousion inthegiven reaction.

Apparatus:Burette,pipette,conicalflask,beakers,burettestand,glazedtile,wash bottle.

Chemicals: Mohr's salt solution, 0.02MKMnO$_4$solution,dilute sulphuric acid.

Principle:Potassiumpermanganateinpresenceofdilutesulphuricacid oxidizesferrous sulphate to ferric sulphate. Ammonium sulphate does not take part in the reaction.

$FeSO_4(NH_4)_2SO_46H_2O \rightarrow FeSO_4+(NH_4)_2SO_4+6H_2O$

$2KMnO_4 +3H_2SO_4 \rightarrow 2MnSO_4+ 3H_2O +5(O)$

$10FeSO_4 +5H_2SO_4+5(O) \rightarrow 5Fe_2(SO_4)_3+5H_2O$

$2KMnO_4+10FeSO_4+8H_2SO_4- \rightarrow K_2SO_4+2MnSO_4+5Fe_2(SO_4)_3+8H_2O$

2moleofKMnO$_4$=10moles ofMohr'ssalt

Formulae:

$M_1V_1/n_1=M_2V_2 /n_2$

WhereM$_1$= Molarity ofKMnO$_4$solution.

V$_1$= volume of KMnO$_4$solution

n$_1$=numberofmolesofKMnO$_4$ M$_2$

= Molarity of Mohr's salt

V$_2$=volumeofMohr's salt

n$_2$=numberofmoles ofMohr's salt

AmountofFerrous ammoniumsulphatepresentinonelitreofgivensolution

=MolarityofMohr'ssaltxGramMol.WtofMohr's salt

Procedure :

1. Rinseand filltheburettewith thegiven KMnO$_4$solution.
2. Pipetteout20mlofMohr's saltsolutionintoacleanconicalflask.
3. Addequalvolumeof dilutesulphuric acid.
4. Titratethesolutionagainst0.02M KMnO$_4$solutiontillpalepinkcolourappears.
5. Notethevol.ofKMnO$_4$used.Itis calledEnd point.
6. Repeatthetitrationuntiltheconcurrentreadingsareobtained.
7. CalculatethemolarityofMohr'ssaltsolutionandamountofMohr'ssaltpresentin one litre of solution by using the above mentioned formulae.

Observations&Calculations:

S.No	Vol.OfMohr's salt solution	Burettereading		Vol. of KMnO$_4$ rundown(b-a)ml
		Initial(a)	Final (b)	
1				
2				
3				
4				

$M_1V_1/n_1 = M_2V_2/n_2$

WhereM$_1$ = Molarity ofKMnO$_4$ solution.

V$_1$= volume of KMnO$_4$ solution

n$_1$=numberofmolesofKMnO$_4$ M$_2$ =

Molarity of Mohr's salt

V$_2$=volumeofMohr's salt

n$_2$=numberofmoles ofMohr's salt

AmountofFerrous ammoniumsulphatepresentinonelitreofgivensolution
=MolarityofMohr'ssaltxGramMol.WtofMohr's salt

=‑‑‑‑‑‑‑‑‑x 392= ‑‑‑‑‑‑‑‑‑‑ g/litre.

ThereforetheamountofMohr'ssaltpresentinonelitreofgivensolution=‑‑‑‑‑‑‑‑‑‑‑‑‑‑‑‑‑‑‑‑‑‑ g/litre.

Result :

TheamountofMohr'ssaltpresentinonelitreofgivensolution=‑‑‑‑‑‑‑‑‑‑‑‑‑‑‑‑‑‑‑‑‑‑ g/litre.

VIVAQUESTIONS:

1. WhatisMohr'ssaltsolution?
2. Write theoxidationreactioninvolvedintheexperiment?
3. WhatisoxidationstateofChromiumionin$K_2Cr_2O_7$?
4. Definenormality?
5. Mentiontheconditionsmaintainedduringtitration.

EXPERIMENTNO-16

DeterminationofSaltConcentrationbyIonexchangeResin

Objective: To determine the $NaNO_3$ salt concentration by Ion-exchange Resin.

Outcome: Thestudentshallbeabletoidentify the ionexchangereactions&roleofion exchange resins in purification of hard water.

Apparatus:-Separatoryfunnel-250ml,columnchromatographictubes-2,glasswoolor sinteredglass disc, beaker ,conical flask , Pipette.

ChemicalsRequired:CationExchangeResin, AnionexchangeResin,$NaNO_3$,NaOH-0.1N, Phenolphthalein , $AgNO_3$-0.1 N, Potassium Chromate

Principle:

Manysubstancesbothneutralandartificialhaveion-exchangeproperties.Allionexchangers have common properties.

a) Theyare almost insoluble inwaterandorganicsolvents.

b) Theycontainactiveor counter ionsthatwillexchangereversiblywithother ioninthe surrounding solution without any appreciable physical change in the material.

c) The ion-exchanger is of complex nature , infact polymeric that carries an electric charge thatisexactlyneutralizedbythe changesonthecounterion. Iftheactiveionsarecations it is called cation exchanger.

Forexamplewidelyusedcationexchangingresinis obtainedbythecopolymerizationof styrene and divinylbenzene followed bysulphonation to give cation exchanging resin. The general representation is RH^+ . where C^+= cation.

$$RH^+ + C^+ \longrightarrow RC^+ + H^+$$

Theanion exchanger isaploymerthatcontainsamineorquarternaryammoniumorOH$^-$ groups in the intergral parts of polymer lattice which is generally represented by R^1OH and the exchange take place as follows

$$R^1OH + A^- \longrightarrow R^1A^- + OH^-$$

Procedure:

Weighout exactly0.5gms airdriedand transferinto acolumnwiththehelpofsmallcamel – hair brush , through afunnelinto thecolumn. Add sufficientdistilled water to cover theresin .Dis lodge the air bubbles that strick to resin by applying intermittent pressure to the rubber tubing.Adjustthe leveloftheoutertube,sothatthe liquidin thecolumnwilldrain1cmabove

resinbeads.Takea250mlseparatoryfunnelwith100mlofthegivenNaNO₃saltsolutionand run this solution at a rate of 2ml per minute. Collect the effluent with standard 0.1 NNaOH using phenolphthalein as indicator. Pink colur is the end point of the solution.

$$R\text{-}H^+ + NaNO_3 \longrightarrow R\text{-}Na^+ + HNO_3$$

$$HNO_3 + NaOH \longrightarrow NaNO_3 + H_2O$$

Take1gm anionexchangein acolumnandtake100mlofthegivensodiumnitratesolutioninto a separatory funnel and add slowly $NaNO_3$ salt solution at rate of 2 ml per minute . Collect the effluent and titrate with 0.1 N Silver nitrate solution using Potassium chromate as indicator

Result:-

Theconcentration and amountof $NaNO_3$salt= _____

Observation&Calculations:-

Cation exchanger column:-

$N_1V_1 = N_2V_2$

V_1 = Volume of Std NaOH=

N_1= Normality of Std NaOH =

N_2=NormalityofNaeffulent= V_2

= Volume of Na effulent =

$N_2 = \dfrac{N_1V_1}{V_2}$

Amount of $Na^+ = N_2 \times 23$ = _____gms/100ml

$N_1V_1 = N_3V_3$

V_1 = Volume of Std $AgNO_3$ =N_1=

Normality of Std $AgNO_3$

=N_2=NormalityofNO₃⁻effluent=

V_2 = Volume of NO_3^- effluent =

$N_3 = N_1V_1/V_3$

Amountof $NO_3^- = N_3 \times 62$ = _____gms /100ml

VIVAQUESTIONS:

1. WhatisIon-exchangeresin?

2. Inestimation,what indicatorsare employed?

3. Whatistheprinciple involvedingiven experiment?

4. Whatis thecolourchangeatendpointofcationexchangereffluent?