East Point College of Pharmacy

East Point Campus, Jnana Prabha, Virgo
Nagar PostBengaluru – 560049, Karnataka

Approved
by
Pharmacy Council of India, New Delhi

Affiliated
to
Rajiv Gandhi University of Health Sciences
Karnataka
Bengaluru – 560041 India

LAB MANUAL

PHYSICAL PHARMACEUTICS-I

B. PHARM 3rd SEMESTER

EAST POINT COLLEGE OF PHARMACY
East Point Campus, Jnana Prabha, Virgo Nagar Post,
Bengaluru – 560049, Karnataka

B Pharmacy

Program Outcomes (PO's)

PO 1- Pharmacy Knowledge

Possess knowledge and comprehension of the core and basic knowledge associated with theprofession of pharmacy, including biomedical sciences; pharmaceutical sciences; behavioral, social, and administrative pharmacy sciences; and manufacturing practices.

PO 2- Planning Abilities

Demonstrate effective planning abilities including time management, resource management,delegation skills and organizational skills. Develop and implement plans and organize workto meet deadlines.

PO 3- Problem analysis

Utilize the principles of scientific enquiry, thinking analytically, clearly and critically, whilesolving problems and making decisions during daily practice. Find, analyze, evaluate and apply information systematically and shall make defensible decisions

PO 4- Modern tool usage

Learn, select, and apply appropriate methods and procedures, resources, and modernpharmacy-related computing tools with an understanding of the limitations.

PO 5- Leadership skills

Understand and consider the human reaction to change, motivation issues, leadership and team-building when planning changes required for fulfillment of practice, professional and societal responsibilities. Assume participatory roles as responsible citizens or leadership roles when appropriate to facilitate improvement in health and wellbeing.

PO 6- Professional Identity

Understand, analyse and communicate the value of their professional roles in society (e.g.health care professionals, promoters of health, educators, managers, employers, employees).

PO 7- Pharmaceutical Ethics

Honor personal values and apply ethical principles in professional and social contexts. Demonstrate behaviour that recognizes cultural and personal variability in values,communication and lifestyles. Use ethical frameworks; apply ethical principles while making decisions and take responsibility for the outcomes associated with the decisions

PO 8- Communication

Communicate effectively with the pharmacy community and with society at large, such as,being able to comprehend and write effective reports, make effective presentations and documentation, and give and receive clear instructions

PO 9- The Pharmacist and society

Apply reasoning informed by the contextual knowledge to assess societal, health, safety andlegal issues and the consequent responsibilities relevant to the professional pharmacy practice.

PO 10- Environment and sustainability

Understand the impact of the professional pharmacy solutions in societal and environmentalcontexts, and demonstrate the knowledge of, and need for sustainable development.

PO 11- Life-long learning

Recognize the need for, and have the preparation and ability to engage in independent and life-long learning in the broadest context of technological change. Self-access and use feedback effectively from others to identify learning needs and to satisfy these needs on an ongoing basis.

Programme Specific Outcomes (PSO's)	
PSO 1	Acquire a thorough foundational knowledge in pharmaceutical sciences, including pharmacology, pharmaceutics, medicinal chemistry, and pharmacognosy, to excel in further academic pursuits
PSO 2	Gain expertise in the application of contemporary pharmaceutical techniques and technologies, enhancing employability across various sectors including the pharmaceutical industry, academia, and research institutions.
PSO 3	Equip with entrepreneurial skills and knowledge of pharmaceutical business management, including market analysis, product development, regulatory affairs, and financial planning, to initiate and run successful ventures in the pharmacy sector

Course Outcomes (CO's)	
Code: BP308P PHARMACEUTICAL ENGINEERING	
CO 1	To know the principles and theory behind the physicochemical properties of drug molecules
CO 2	Determination of PKa partition co-efficient, HLB, stability constant and adsorption studies
CO 3	Determination of CST, surface tension and CMC
CO 4	To understand the importance of physicochemical properties in developing formulation of new drugs

EAST POINT COLLEGE OF PHARMACY
East Point Campus, Jnana Prabha, Virgo Nagar Post,
Bengaluru – 560049, Karnataka

Table of Contents

Recommended Books: (Latest Editions)

1. Physical pharmacy by Alfred Martin
2. Experimental pharmaceutics by Eugene, Parrott.
3. Tutorial pharmacy by Cooper and Gunn.
4. Stocklosam J. Pharmaceutical calculations, Lea & Fibiger, Philadelphia.
5. Liberman H.A, Lachman C., Pharmaceutical Dosage forms, Tablets, Volume-1 to 3, Marcel Dekker Inc.
6. Liberman H.A, Lachman C, Pharmaceutical dosage forms. Disperse systems, volume 1, 2, 3. Marcel Dekker Inc.
7. Physical pharmaceutics by Ramasamy C and Manavalan R.
8. Laboratory manual of physical pharmaceutics, C.V.S. Subramanyam, J. Thimma settee

EAST POINT COLLEGE OF PHARMACY
East Point Campus, Jnana Prabha, Virgo Nagar Post,
Bengaluru – 560049, Karnataka

Experiment No. 01

DETERMINATION OF SOLUBILITY OF DRUG AT ROOM TEMPERATURE

Aim: To determine the solubility of benzoic acid at different temperatures.

Requirements: benzoic acid, distilled water, 0.1N Sodium hydroxide, phenolphthalein indicator, and filter paper, Measuring cylinder, funnel, beaker, conical flask, 10 ml bulb pipette, rubber bulb, burette, burette stand.

Principle:

The amount of drug dissolved in solution at a particular temperature is called solubility. Example: The solubility of paracetamol is 1 g in 70 ml water at 20 ^{0}C. The solubility of a drug is determined by preparing a saturated solution of the drug. A saturated solution is prepared by shaking excess quantity of the drug with the solvent for a long time (48 hours). This system is filtered and the saturated solution is analyzed for drug content by titration or suitable analytic method. In this experiment solubility of benzoic acid is determined by using distilled water. The amount of benzoic acid dissolved in the solvent is analyzed by titrating with 0.1 N Sodium hydroxide solution using phenolphthalein as indicator. When a drug (benzoic acid) has poor solubility in water, then the solubility of benzoic acid is improved by rise of temperature.

Procedure:

1. Take 50 ml of distilled water into a 100 ml beaker. Add required quantity of benzoic acid andshake vigorously for 30 minutes. If the added benzoic acid has dissolved, add further some amount of benzoic acid and continue shaking to obtain a saturated solution.
2. Heat the benzoic acid on the water bath up to 85^{0}C.
3. Allow the temperature to fall gradually to 80^{0}C.
4. Filter the contents into a clean dry beaker.
5. Titrate 10 ml of the filtrate with 0.1 N sodium hydroxide solution using phenolphthalein asindicator.
6. Continue the procedure and obtain data of solubility at 70,60,50,40 and 30^{0}C temperatures.

Draw a plot by taking solubility of benzoic acid on y-axis and temperature on x-axis.

Calculate the solubility of benzoic acid in water

Observations and Calculations:

Sl. No	Temperature (^{0}C)	Volume of sodium hydroxide consumed (ml) (V1)	Normality of benzoic acid (N2)	Solubility of benzoic acid (gm/ml)
1.	80			
2.	70			
3.	60			
4.	50			
5.	40			
6.	30			

Equivalent weight of benzoic acid is 122 gm

Normality of sodium hydroxide (N_1) is 0.1N Volume of sodium hydroxide consumed is (V_1)

Volume of benzoic acid (V_2) is 10 ml sample taken at different temperatures.

$$\text{Normality of benzoic acid } (N_2) = \frac{N_1 V_1}{V_2}$$

$$\text{Solubility of benzoic acid} = N_2 \times \frac{122}{10}$$

Solubility of the drugs is expressed in various units in Merk Index

Term	Parts of solvent required for 1 part of solute
Very soluble	Less than 1part
Freely soluble	1 to 10 parts
Soluble	10 to 30 parts
Sparingly soluble	30 to 100 parts
Slightly soluble	100to 1000 parts
Very slightly soluble	1000 to 10,000 parts
Practically insoluble	More than 10,000 parts

Report: The solubility of benzoic acid in water_____gm/ml at 80^{0}C.As the temperatureincreases the solubility of benzoic acid is increased.

Experiment No. 02

DETERMINATION OF P^{Ka} VALUE BY HALF NEUTRALISATION/ HENDERSON HASSEL BALCH EQUATION

Aim: To determine the P^{Ka} value of the weak acid (acetic acid) by Henderson Hassel Balch equation.

Requirements: Acetic acid, distilled water, 0.1N Sodium hydroxide, 0.1N Oxalic acid, Measuring cylinder, funnel, beaker, conical flask, pH meter.

Principle:

pH is a measure of hydrogen ion concentration, a measure of the acidity or alkalinity of a solution. The pH scale usually ranges from 0 to 14. Aqueous solutions at 25°C with a pH less than seven are acidic, while those with a pH greater than seven are basic or alkaline. A pH level of is 7.0 at 25°C is defined as 'neutral' because the concentration of H_3O^+ equals the concentration of OH^- in pure water. pH is given by equation as

$$pH = -\log[H^+]$$

where log is the base-10 logarithm and $[H^+]$ stands for the hydrogen ion concentration in units of moles per liter solution. pH can be measured by using Henderson Hassel Balch equation which is given by

$$pH = pKa + \log \frac{(salt)}{(acid)}$$

To derive the dissociation constant (pKa). Consider that the weak acid under gone partial dissociation.

$$CH_3COOH + H_2O \leftrightarrow CH_3COO^- + H_3O^+$$

At equilibrium $K = \frac{K1}{K2} = \frac{(CH_3COO^-)\ (H_3O^+)}{(CH_3COOH)\ (H_2O)}$

where K1 and K2 are the rate constants of forward and backward reactions respectively. (H_2O) is a constant at about 55.3 moles/liter.

$$Ka = K \times 55.3 = \frac{(CH_3COO^-)\ (H_3O^+)}{(CH_3COOH)}$$

where Ka is the dissociation constant. $(H_3O^+) = \frac{Ka\ (CH_3COOH)}{(CH_3COO^-)}$

Take –log on both sides, it becomes as

$$-\log (H_3O^+) = -\log K_a - \log (CH_3COOH) + \log (CH_3COO^-)$$

$$pH = pK_a + \log \frac{(CH_3COO^-)}{(CH_3COOH)}$$

$$pH = pK_a + \log \frac{(salt)}{(acid)}$$

In this study pH, pK_a and K_a will be determined for acetic acid.

Procedure:

Prepare the buffer solutions using standard buffer tablets 4, 7 and 9.4. Calibrate the pH meter by using the buffer solutions. Take 0.05 ml of acetic acid in volumetric flask having capacity of 100 ml and make up the volume. This solution was taken in beaker and measures the pH of the solution. Finally calculate pK_a of acetic acid by using the equation.

Observations and Calculations:

Molecular weight of acetic acid = 60.05Weight per ml of solution = 1.0495

$pH = -\log[H^+]$

$$K_a = (CH_3COO^-)\ (H^+) = (H^+)^2\ \frac{(CH_3COOH)}{(CH_3COOH)}$$

60.05 gm of acetic acid in 1000 ml = 1M

6.005 gm of acetic acid in 100 ml = 1M

(CH_3COOH) = Volume of acetic acid x weight per ml = 0.05 x 1.0495 = 0.0087 moles per liter.
Weight of acetic acid for 1M 6.005

$$K_a = \frac{(10^{-pH})^2}{(CH_3COOH)}$$

Therefore $pK_a = pH = -\log [H^+]$

Report:

The pH of the acetic acid solution is ----------

The dissociation constant (K_a) of acetic acid is ----------

The pK_a of the acetic acid solution is----------

Experiment No. 03

DETERMINATION OF PARTITION COEFFICIENT OF BENZOIC ACIDBETWEEN BENZENE AND WATER

Aim: To determine the partition coefficient of benzoic acid between benzene and water.

Requirements: Benzene, benzoic acid, 0.1N sodium hydroxide solution, phenolphthalein indicator, separating funnel, tripod stand, reagent bottles, two small beakers, measuring cylinder, conical flask, burette, burette stand, tile and digital balance.

Principle:

When a substance is added to a system containing two immiscible liquids, it distributes between the two liquids in a definite ratio." This is called Nernst distribution law. The added substance should have solubility in the two liquids for distribution to occur. This is known as the partition coefficient **K** of a substance between two liquids is given by the formula

$$\mathbf{K} = \frac{\text{Concentration of substance in organic layer}}{\text{Concentration of substance in aqueous layer}} = \frac{C1}{C2}$$

In the present experiment, distribution of benzoic acid between benzene and water is studied. Benzoic acid is an organic substance and has high solubility in benzene. It has less solubility in water. As a result, benzoic acid will partition preferably into benzene layer. The formula used for calculating partition coefficient of benzoic acid between benzene and water is given below. C_1 and C_2 are concentration of benzoic acid in organic and aqueous layer. In the present experiment, benzoic acid is shaken with benzene and water for 30 minutes to achieve distribution. Shaking is required to achieve distribution equilibrium. At equilibrium the speed of forward process is equal to the speed of backward process.

Benzoic acid is distributed as associated molecules in benzene layer and un associated molecules in aqueous layer. Hence the equation is given as follows.

$$K=\sqrt{\frac{Concentration\ of\ substance\ in\ organic\ layer}{Concentration\ of\ substance\ in\ aqueous\ layer}}=\sqrt{\frac{C1}{C2}}$$

The partition coefficient K will be remains constant only if there is neither association nor dissociation of solute molecules in both the phases.

Procedure:

Preparation of 0.1N Sodium hydroxide: 4 gm of sodium hydroxide was dissolved in 1000 ml of distilled water and make up the final volume in volumetric flask.

1. Weigh the samples (250 mg, 500 mg and 750 mg) of benzoic acid into three reagent bottlesand add 50 ml of benzene and 50 ml of water to all the three reagent bottles.
2. Keep the bottles on constant temperature water bath and Shake the bottles for 30 minutes.
3. Transfer the contents into a separating funnel and allow them to separate as two layers.
4. Collect the aqueous layer and titrate 10ml of sample with 0.1N sodium hydroxide solutionusing phenolphthalein as indicator.
5. Similarly collect the organic layer (benzene) and titrate 10ml of sample with 0.1N sodiumhydroxide solution using phenolphthalein as indicator.
6. Calculate the partition coefficient of benzoic acid between benzene and water.

Observations and Calculations:

Equivalent factor: Each ml of 0.1N sodium hydroxide = 0.0122 gm of benzoic acid

Concentration of benzoic acid = Volume of sodium hydroxide consumed x 0.0122

S.NO	Volume of aqueous / benzene layertaken	Volume ofsodium hydroxide consumed in ml	Concentrationo benzoic acid	$\sqrt{C1}$	Partition coefficient = $\frac{\sqrt{C1}}{C2}$
1	10 ml organic		C1=		
2	10 ml organic		C1 =		
3	10 ml aqueous		C2 =		
4	10 ml aqueous		C2 =		

Report: The partition coefficient of benzoic acid between benzene and water is____.

Experiment No. 04

DETERMINATION OF PARTITION COEFFICIENT OF IODINE BETWEEN CARBON TETRA CHLORIDE AND WATER

Aim: To determine the partition coefficient of iodine between carbon tetra chloride and distilled water.

Requirements: Iodine, carbon tetra chloride, 0.1N sodium thiosulphate solution, 0.005N sodium thiosulphate solution, starch mucilage as indicator, separating funnel, tripod stand, reagent bottles, two small beakers, measuring cylinder, conical flask, burette, burette stand, tile and digital balance.

Principle:

When a substance is added to a system containing two immiscible liquids, it distributes between the two liquids in a definite ratio." This is called Nernst distribution law. The added substance should have solubility in the two liquids for distribution to occur. This is known as the partition coefficient **K** of a substance between two liquids is given by the formula

$$\mathbf{K} = \frac{\text{Concentration of substance in organic layer}}{\text{Concentration of substance in aqueous layer}} = \frac{C_1}{C_2}$$

Where K is known as partition coefficient or distribution coefficient, C_1 and C_2 are the total concentrations of the solute in the two layers of organic and aqueous phases.

Procedure:

Preparation of saturated solution of Iodine: Dissolve the sufficient amount of iodine in carbon tetra chloride until some solid remains undissolved.

Preparation of 0.1N sodium thiosulphate solution: 26 gm of sodium thiosulphate and 0.2 gm of sodium carbonate was dissolved in 1000 ml of distilled water and make up the final volume in volumetric flask.

Preparation of 0.005N sodium thiosulphate solution: 1.3 gm of sodium thiosulphate and 0.01 gm of sodium carbonate was dissolved in 1000 ml of distilled water and make up the final volume in volumetric flask.

1. Bymeans of a graduated pipette place about 30ml and 15ml of a saturated solution of iodine in carbon tetra chloride was prepared (stock solution) and properly labeled for glass stoppered bottles.
2. To these bottles add 100 ml of distilled water and shake the bottles for 20 minutes while keeping in water bath at room temperature. Keep it aside and allow them to separate as two phases of solution.
3. Withdraw 10 ml of the organic layer from first bottle carefully and titrate against 0.1 N sodium thiosulphate using starch solution as indicator.
4. Withdraw 10 ml of the organic layer from second bottle carefully and titrate against 0.1 N sodium thiosulphate using starch solution as indicator.
5. Similarly withdraw 10 ml of the aqueous layer from first bottle carefully and titrate against 0.005 N sodium thiosulphate using starch solution as indicator.
6. Similarly withdraw 10 ml of the aqueous layer from second bottle carefully and titrate against 0.005 N sodium thiosulphate using starch solution as indicator.
7. Calculate the partition coefficient of Iodine between carbon tetra chloride and water.

Observations and Calculations:

Titration of organic layer

Sl. No	Container	Volume of 0.1N sodium thiosulphateconsumed in ml V1	Concentration of iodinein organic layer (N2= N1V1/ V2)
1	Bottle 1		
2	Bottle 2		

N1= Normality of the sodium thiosulfate = 0.1N V1= volume of the sodium thiosulfate consumed =?V2= Volume of the organic layer = 10 ml

N2= Normality (concentration) of the iodine =

Titration of aqueous layer

Sl. No	Container	Volume of 0.005Nsodium thiosulphateconsumed in Ml(V1)	Concentration of iodinein aqueous layer N2= N1V1/ V2
1	Bottle 1		
2	Bottle 2		

N1= Normality of the sodium thiosulphate = 0.005N

V1= volume of the sodium thiosulphate consumed =?

V2= Volume of the aqueous layer = 10 ml

N2= Normality (concentration) of the iodine =?

For bottle 1: K = $\frac{\text{Concentration of substance in organic layer}}{\text{Concentration of substance in aqueous layer}} = \frac{C_1}{C_2}$

For bottle 2: K = $\frac{\text{Concentration of substance in organic layer}}{\text{Concentration of substance in aqueous layer}} = \frac{C_1}{C_2}$

Report: The partition coefficient of iodine between carbon tetra chloride and distilled water was found to be---------- --.

Experiment No. 05

DETERMINATION OF % COMPOSITION OF SODIUM CHLORIDE IN SOLUTION USING PHENOL WATER SYSTEM BY CST METHOD

Aim: To determine the % composition of sodium chloride in a solution using phenol water system by CST method.

Requirements: Phenol, distilled water, sodium chloride, thermometer, pipette, beaker, water bath and funnel.

Principle:

The temperature at which complete miscibility is reached as the temperature is raised or in some cases lowered used of two liquids that are partially miscible under ordinary conditions called also consulate temperature. The lower critical solution temperature (CST) or lower consulate temperature is the critical temperature below which the components of a mixture are miscible for all compositions. The word lower indicates that the LCST is a lower bound to a temperature interval of partial miscibility, immiscibility for certain compositions only. For example, the system triethylamine water has an LCST of 19^0 C, but not at higher temperatures. The Upper critical solution temperature or upper consulate temperature is the critical temperature above which the components of a mixture are miscible in all proportions. The word upper indicates that the UCST is an upper bound to a temperature range of partial miscibility, or miscibility for certain compositions only. For example, hexane nitrobenzene mixtures have a UCST of 19^0C, so that these two substances are miscible in all proportions above 19^0C but not at lower temperatures. When water and phenol are mixed together two layers are formed. The upper layer is solution of phenol in water. At a given temperature, composition of each solution is fixed and both solutions are in equilibrium. The two solution of different composition are existing in equilibrium with one another are known as conjugate solution. As the temperature increases, mutual solubility increases at a particular temperature this conjugate solution becomes completely miscible with one another. A temperature of which two conjugate solution are mutually soluble is called miscibility temperature. The miscibility temperature can be identifying as the disappearance of turbidity and reappearance of turbidity.

Procedure:

1. Prepare 50 ml of 1% w/v of sodium chloride in water and this stock solution is used for the preparation of different concentrations such as 0.1, 0.2, 0.4, 0.6, 0.8 and 1% v/v in theexperiment.
2. Take 10 ml of stock solution each in boiling tubes and add 2 ml of phenol to each sample of stock solution.
3. Heat the mixture on water bath and note the temperature at which mixture becomes one layer in all the tubes (turbidity disappears). Note this miscibility temperature as $T_1{}^0C$.
4. Stirrer and thermometer are introduced in the sample tube. Continuously stir and observe the reappearance of turbidity of the mixture after cooling. Note this temperature at which turbidity reappears as $T_2{}^0C$.
5. Take the average of the temperature values that gives the CST of the solution. Similarly take 10 ml of the given unknown sample and add 2 ml of phenol to the sample and determine the CST of the sample.
6. Draw mutual solubility curve by plotting average miscibility temperature on Y-axis and percent composition of sodium chloride on X-axis. It will give the straight line. Using thegraph read the percentage composition of unknown sample.

Observations and Calculations:

0.5 gm of sodium chloride in 50 ml gives 1% sodium chloride solution.

Sl. No	Sodium chloride solution [ml]	Distilled water [ml]	Percentage compositionof sodium chloride	Turbidity disappears temperature [T1]	Turbidity reappears temperature [T2]	Average of temperature
1	1	9	0.1			
2	2	8	0.2			
3	4	6	0.4			
4	6	4	0.6			
5	8	2	0.8			
6	10	0	1.0			
7	unknown	Up to 10	unknown			

Report: The CST of unknown sample was found to be ----------^{0}C and the percent composition of sodium chloride in a solution (from graph) is---------- %.

Experiment No. 06

DETERMINATION OF SURFACE TENSION OF GIVEN LIQUIDS BY DROP COUNT AND DROP WEIGHT METHOD

Aim: To determine the surface tension of the given liquids by drop count and drop weight method

Principle: The behavior of the molecules of the surface of the liquid is different from those in any other position of liquid the molecules present at the surface are always pulled down wards by crystalline force if attraction. The magnitude at this force acting perpendicular tothe plane of the surface is called as surface tension is has the dimension forces length and is expressed in newton/meter (CGS units dyne/cm). Surface or interface tension can be determined by several methods. Namely

1. Drop formation method
2. Capillary rise method
3. Bubble pressure method
4. DuNouy or ring detach method

The choice of the perpendicular method depends on the weather surface or interface tension to be determine the accuracy and convenience. Size of the sample drop plate method is usefulfor relative dimension.

Procedure:

Determination of density

1. The weight of an empty specific gravity bottle was determined.
2. The bottle was filled with water and weight is determined.
3. The water was replaced by with given liquid and the weight was determined.
4. The density of liquid calculating using the formula.

Density of the unknown liquid = ρ = Mass/Volume

Determination of surface tension by drop count method

1. The stalagmometer was clean to remove any grease and rinsed with alcohol and dried.
2. The meter was filled with water above the mark A. the no. of drops of water whenit flows between marks A to B was counted. The procedure was repeated in duplicate and average no. of drops was calculated.

3. The water was removed and the experiment was repeated as above. The average no. of drops of liquid was calculated.
4. The surface tension of the given liquid was determined using formula.

Surface tension of liquid,

$$\gamma 2 = \frac{\gamma 1 \times n1 \times P2}{n2\rho 1}$$

Where,

$\gamma 1$ = surface tension of water

γ_2 =Surface tension of liquid

$\rho 1$ = density of unknown liquid

P2 = density of known liquid

n_1 = Mean number of drops obtained for water

n2= Mean number of drops of obtained for liquid

Determination of surface tension by drop weight method

1. The stalagmometer was clean to remove any grease and rinsed with alcohol anddried.
2. In a weighing bottle about 20 drops of liquid are received from stalagmometer andweighed.
3. Thus, weight of one drop is found.
4. Repeat the procedure for second liquid (reference, usually water) and weight of onedrop determined as before.
5. Using following equation one can calculate the surface tension of unknown liquid.

OBSERVATION AND CALCULATION:

Weight of empty specific gravity bottle (w1) = Weight of specific gravity bottle + sample (w2)

=Weight of sample (W_3) = (W_2-W_1) =

Density of the sample = _____

Sl. No	Sample	No of drops		Average
		Trial 1	Trial 2	
1	Water			
2	Sample			

Report:

1. The surface tension of the given liquids by Drop count method was found to be _____Dyne/cm.

2. The surface tension of the given liquids by Drop weight method was found to be _____Dyne/cm.

Experiment No. 07

DETERMINATION OF HLB NUMBER OF A SURFACTANT BY SAPONIFICATION METHOD

Aim: Determination of HLB number of a surfactant by saponification method.

Requirements: Glyceryl mono stearate (GMS, Surfactant), 0.5 N alcoholic potassium hydroxide solution, stearic acid, ether, 0.5N HCl, 0.1 N NaOH solution, Phenolphthalein indicator. Round bottom flask, Reflux condenser etc.

Principle:

Griffin (1949) developed an arbitrary scale. The HLB value is defined as percentage molecular hydrophilic groups. According to Griffin scale there are 100 such grouping and therefore 20 different HLB values are possible and depending on the HLB value they have their own applications.

HLB of surfactant like polyhydric alcohols and fatty acid esters such as glyceryl monostearate can be calculated using formula.

$$HLB = 20(1-S/A)$$

Where S = Saponification number of the ester, A = acid number of the fatty acid. Saponification number is defined as the number of milligrams of potassium hydroxiderequired to neutralize the acid obtained during saponification of one gram of sample. Acidnumber is defined as the number of milligrams of potassium hydroxide required to neutralizethe free acid in one gram of sample.

Saponification number of many substances like bees wax and wool fat derivatives cannot be easily estimated. In such cases HLB is calculated by following equation.

HLB system provides information regarding the nature and other properties of the surfactant.

Procedure:

Preparation of 0.5N alcoholic KOH

Dissolve 14.025 gms of potassium hydroxide in 500ml of alcohol to get 0.5 N KOH. Prepare this solution a day before and filter if necessary.

0.5N HCl:

Add 43.47 ml of concentrated Hcl in 1000ml of distilled water taken in 1000ml volumetric flask.Add water to1000ml and shake well

0.5N Na2Co3:

Weigh 2.65g of sodium carbonate in 100ml water to get 0.5N Na2Co3

Determination of HLB value:

1.Weigh accurately about 2gms of the surfactant in to a 250ml round bottom flask.

2.Add 25 ml of 0.5N alcoholic solution of potassium hydroxide.

3. Attach a reflux condenser and boil on a waterbath for one hour, frequently rotating the contents of the flask. Porcelain chips is added to avoid bumping.

After saponification, cool and add one ml of phenolphthalein indicator.Titrate the excess alkali with 0.5N Hcl. Note the number of ml required as a.

5. Repeat the experiment with the same quantities of the same reagents in the same manner without the surfactant. Note the number of ml of 0.5N Hcl required as b.

6. Standardize the hcl using sodium carbonate and methyl red as indicator.

Observations:

Saponification value'S'

Weight of the given surfactant w= gms

Blank titration value ,b = ml

Sample titration value a= ml

Saponification value'S' $= \frac{(b-a)\times 0.02804\times 100\times normality\ of\ Hcl}{W\times 0.5}$

Acid value A=

HLB value = 20(1-S/A)

Report:

Experiment No.08

DETERMINATION OF FREUNDLICH AND LANGMUIR CONSTANTS USING ACTIVATED CHARCOAL

Aim: to study the adsorption behavior in acetic acid on charcoal

Principle: Adsorption phenomenon, at solid -liquid interface can be studied using acetic acid & charcoal when a solution of acetic acid in water is shaken in presence of charcoal. As a result the concentration of acid is increased the magnitude of adsorption also increases at a given temperature, this relationship is expressed Freundlich adsorption equation.

$$\frac{x}{m} = Kc(1/m)$$

Where x= concentration of acetic acid adsorbed,N

M= weight of adsorbent used,g

C= concentration of acetic acid in n

K= constant

Above equation gives a plot as shown in figure, equation can be converted into a linear expression by taking on logarithm

$$\log\frac{x}{m} = \log k + \frac{1}{n}\log c$$

Thus plotting log(x/m) on y-axis & equlibrium concentration on x-axis gives straight line. The constant K & n can be obtained from the slope & intercept of line.

Procedure:

Prepare different concentration of acetic acid as given in the below table. Transfer them into conical flask (or reagent bottle,250ml) label them as A-F.

1. Weigh about 2.0g of activated charcoal & prepare 6 such packets
2. Transfer the activated charcoal samples carefully into the acetic acid solution bottle
3. Cork the bottles securely
4. Keep the bottles in a constant temperature water bath (room temperature) & shake them in a shaker bath for half an hour. Ordinary water bath can also be used for intermittent shaking for half an hour. Normally equilibrium is attained remove both mixture for several hours
5. After equilibrium is attained remove the bottle from the constant temperature water bath
6. Filter the acetic acid solution using whatmann paper
7. Pipette out 10ml of filterate into a conical flask
8. Titrate them against 0.1N NaoH solution using phenolpthalein indicator.

9. Plot the graph taking $\log\frac{x}{m}$ +2 on y-axis & log(C+2) on x - axis
10. Report: According to freundlich adsorption equation the constant for adsorption of actic acid on activated charcoal is

Observation & Calculation:

Flask No	Volume of NaoH(Consumed in ml)			Equilibrium conc of acetic acid	Log(C+2)	X=(C_o-C)×M	$\frac{x}{m}$	$\log\frac{x}{m}$+2
	IR	FR	Total					

Report:

Experiment No. 09

DETERMINATION OF CRITICAL MICELLAR CONCENTRATION OF SURFACTANTS

Aim: To determine the CMC of given surfactant by using stalagmometer based on surface tension measurement.

Theory: **S**surfactant is a usually describes as a substance that lower the surface tension of water. However, it requires more precise definition. In physical sense surfactant is defined as a substance which gets preferentially adsorb at the interface and exhibit self-association in thebulk of solution at a specific concentration. Chemically surfactant is defined as a substance offairly high molecular weight and possess polar and non-polar regions for this reason there also called as amphiphiles.

When surfactants are added to water at low concentration the molecules exist independently as monomers as the concentration these aggregates 50 or more monomers.

CMC is defined as concentration range at which micelles are formed by surfactants. CMC is not a point concentration but a range unit are w/v, mole/lit, mole/1000gm etc.,

Micelle formation: Below CMC, surfactants are preferentially adsorbed mainly at air-water interfaces as monomers when these encounter aqueous environment water molecules reject water insoluble tail therefore tails move from water, while head is attached by polar water molecules. As the concentration increases the interface and bulk phase become saturated and concentration is called as CMC. Any further increase in the concentration they lead to formation of micelles.

Applications:

1. Bacterial activity of certain drug is observed to change drastically when given in solubilized system.
2. Micellar solutions are used to prepare liposomes. Which are currently used as a drug delivery system.
3. The penetration of hexyl resorcinol into pinwarm acaris is increased by the presenceof low concentration.

At low concentration surfactant reduces surface tension of water as concentration increase surface tension of liquid is determined by drop count method. Densities water and surfactant by using specific gravity bottle.

Surface tension of liquid, $\gamma_2 = \frac{\gamma 1 \times n1 \times P2}{n2\rho 1}$

Plot a graph taking concentration of surfactant on x – axis surface tension on y- axis initially there is negative slop at CMC. Slope will be zero above CMC surface tension remaining constant.

Procedure:

1. Prepare the 1%w/v of Sodium lauryl sulphate stock solution. And it is prepared into various concentration as 0.05, 0.1, 0.2, 0.5 and 1.0%.
2. The stalagmometer was clean to remove any grease and rinsed with alcohol and dried.

3. The meter was filled with water above the mark A. the no. of drops of water when it flows between marks A to B was counted. The procedure was repeated in duplicate and average no. of drops was calculated.
4. The water was removed and the experiment was repeated as above. The average no.of drops of liquid was calculated. Select a specific gravity bottle 25 ml and note w1fill it with water and record as w2 remove and dry fill it with surfactant solution w3

Density of water is 0.99gm/cc. similarly density of other can be calculated.

Observation:

Conc. of surfactant	No. of drops		Avg no. Of drops	Density kg/m^3	Surface tension N/m
	Trail – I	Trail - II			
0					
0.05					
0.1					
0.2					
0.5					
1.0					

Calculation of surface tension:

Surface tension of water is 72 dyne/cm. similarly calculate surface tension for other solutions.

Take the concentration of Surfactant on x – axis and surface tension on y-axis.

Report: Critical Micellar concentration of given surfactant is .....% w/v

Experiment No. 10

DETERMINATION OF STABILITY CONSTANT AND DONOR ACCEPTORRATIO OF PABA – CAFFEINE COMPLEX BY SOLUBILITY METHOD

Aim: To determine the complex stability constant and donor acceptor ratio of caffeine and para amino benzoic acid (PABA) by solubility method.

Requirements: Volumetric flask, beakers, conical flasks, pipette, burette, funnel, Para amino benzoic acid, sodium hydroxide (0.025N), caffeine, phenolphthalein indicator, Whatman filter paper.

Principle:

Complex compounds are defined as those molecules in which most of the bonding structures can be described by classical theories of valency between atoms or molecules. Complexes possess some properties, which are different from those of its components. Properties such as solubility, light absorption, conductance, partitioning behavior and chemical reactivity are studied to confirm the formation of complexes. For example, para Amino Benzoic acid and caffeine form complexes in solution. This results in enhanced solubility of PABA at low concentrations of caffeine. Further increase in concentration of caffeine results in decreased solubility of PABA. Therefore, the change in the solubility profile is taken as a criterion to decide the complexation behavior. The equation for the formation of complex is

$$\text{PABA} + Caffeine \rightarrow PABA - caffeine$$

The interaction may be due to dipole-dipole force or hydrogen bonding between the polar carbonyl groups of caffeine and hydrogen atom of the acid. The secondary interaction may probably occur between the nonpolar parts of the molecules. The analysis of complexes generally involves the estimation of two parameters. These are represented by equations as

$$\text{Stoichiometric ratio} = \frac{[\text{caffeine in complex}]}{[\text{PABA in complex}]}$$

$$\text{Complex stability constant} = \frac{[\text{PABA}-\text{caffeine}]}{[\text{PABA}][\text{caffeine}]}$$

In this method, caffeine is taken in different concentrations in a series of flasks. Excess quantity of PABA (same quantity) is added to all the flasks. These flasks are corked and agitated at a constant temperature bath, until equilibrium is attained. The samples are filtered and saturated

solution is collected and analyzed for drug content. The corresponding concentrations are substituted in equations 1 and 2.

Procedure:

Caffeine stock solution (0.1N): Weigh 1.949 gm of anhydrous caffeine and transfer into 100 mlof volumetric flask and add distilled water to make up final volume.

Para amino benzoic acid (PABA): Weigh accurately the required number of samples containing 200 mg of Para amino benzoic acid.

Preparation of 0.025 N sodium hydroxide: 1 gm of sodium hydroxide was dissolved in 1000 ml of distilled water and make up the final volume in volumetric flask.

1. Prepare various concentrations of caffeine (use 100ml conical flasks or beaker). The concentrations of caffeine are given in table. Transfer the samples of PABA into each flask containing the above caffeine solutions. Fix the flasks in a constant temperature bath and shake them for 30 minutes to attain equilibrium.
2. Filter the above solutions with Whatman filter paper and 10 ml of filtrate was taken and titrated with 0.025N sodium hydroxide using phenolphthalein indicator. Complete the titration of all samples and process the data in the table.
3. Draw a plot between concentration of caffeine on X-axis and concentration of PABA on Y-axis. Calculate the complex stability constant and donor acceptor ratio of caffeine and para amino benzoic acid (PABA) using the equations.

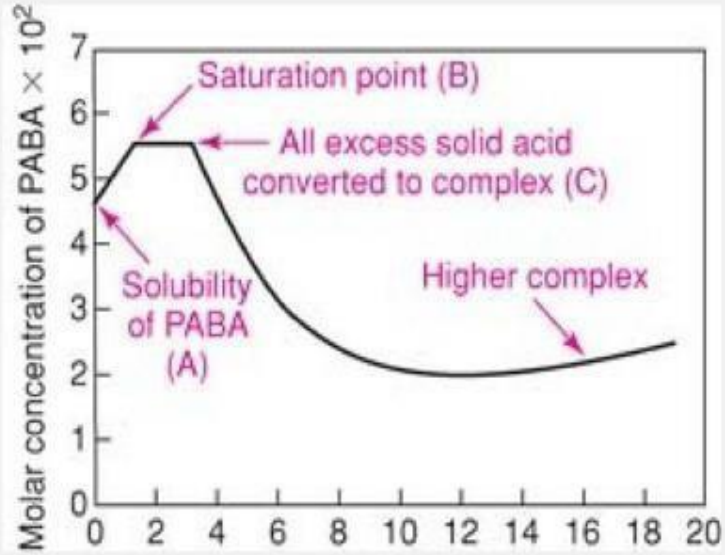
Observations and calculations:

Concentration of caffeine solution

S. No	Caffeine solution (ml)	Distilled water (ml)	Concentration of caffeine mol/liter
1	0	20	0
2	2	18	1
3	4	16	2
4	6	14	3
5	8	12	4
6	10	10	5
7	12	8	6
8	16	4	8

Analysis of complex:

S. No	Concentration of caffeine mol/liter	Volume of sodium hydroxide consumed in ml	Concentration PABA mol/liter
1	0		
2	1		
3	2		
4	3		
5	4		
6	5		
7	6		
8	8		



The solubility of *para*-aminobenzoic acid (PABA) in the presence of caffeine.

Report: The complex stability constant of caffeine and para amino benzoic acid (PABA) is----
The donor acceptor ratio of caffeine and para amino benzoic acid (PABA) is --.

Experiment No. 11

DETERMINATION OF STABILITY CONSTANT AND DONOR ACCEPTORRATIO OF COPPER-GLYCINE COMPLEX BY pH TITRATION METHOD

Aim: To determine the complex stability constant (log β) and donor acceptor ratio (n) of Copper

- Glycine complex pH by titration method.

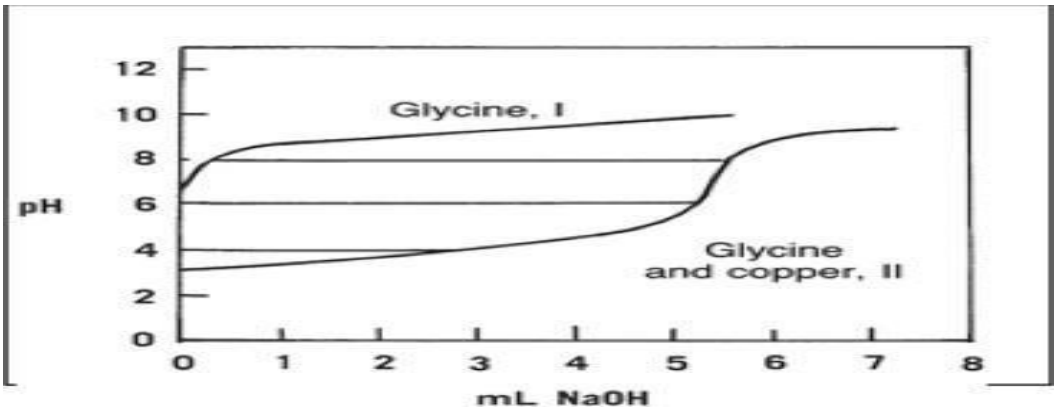
Requirements: Volumetric flask, beakers, conical flasks, pipette, burette, funnel, cupric chloride Glycine, sodium hydroxide (0.25N), phenolphthalein indicator, pH meter, buffer tablets, pH 7, 4 and 9.4.

Principle:

Complexation of copper ions with Glycine can be represented by the following equation.

$$Cu^{+2} + 2NH_3CH_2COO^- \rightarrow Cu^{+2} + (NH_2CH_2COO^{-)}\ 2 + 2H^+$$

Because of two protons are formed in the reaction of equation the addition of glycine to a solution containing cupric ions should result in a decrease in pH. Titration curves can be obtained by adding a strong base to a solution of glycine and to another solution containing glycine and a copper salt and plotting the pH against the equivalents of base added. The results of such a potentiometric titration are shown in the figure. The curve for the metal–glycine mixture is well below that for the glycine alone, and the decrease in pH shows that complexation is occurring throughout most of the neutralization range.



Titration of glycine and cupric glycine complex solution. The difference in pH for a given quantity of base (sodium hydroxide) added indicates the occurrence of a complex.

The average number of ligand groups bound per metal ion can be given by equation as

$$n = \frac{\text{Total concentration of ligands bound}}{\text{Total concentration of metal ion}}$$

The horizontal distance represents the amount of alkali added in the titration. This quantity is equals to the concentration of ligand bound to metal at any pH. The total concentration of metal ion taken initially is known. Thus, n can be calculated. The stability constant (β) and pH of free glycine are related as

$$p(A) = ½ \log \beta \text{ at } n = 1$$

p(A) can be estimated using the equation

$$p(A) = pKa - pH - \log([HA]_{initial} - [NaOH])$$

Where pK_a is dissociation constant of glycine, (9.69)

[NaOH] is concentration of sodium hydroxide in mol/lit.

Procedure:

Preparation of 0.25N sodium hydroxide: 10 gm of sodium hydroxide was dissolved in 1000 ml of distilled water and make up the final volume in volumetric flask.

Preparation of Glycine solution (3.34×10^{-2}mol/lit): Weigh 250 mg of glycine and transfer into a 100 ml of volumetric flask, add distilled water and make up the volume.

Complex solution (Glycine – 3.34×10^{-2}mol/lit; cupric chloride –9.45×10^{-3}mol/lit): Weigh 250 mg of glycine and160 mg of cupric chloride and transfer into a 100 ml of volumetric flask, add distilled water and make up the volume. Prepare two such samples.

Kinetic method:

Transfer the 75 ml of glycine solution into a beaker. Measure the pH of the solution. Gradually add the 0.25N of sodium hydroxide solution to the glycine solution.

1. Transfer the 75 ml of glycine-cupric complex solution into a beaker. Measure the pH of the solution. Gradually add the 0.25N of sodium hydroxide solution to the glycine-cupric complex solution.
2. Identify the range where the sudden increase in pH is obtained in the complex solution. Take another sample of 75 ml of complex solution and add 1 ml increment up to 5 ml to the complex mixture and report the data.
3. Titrate the complex solution further (note: If sudden increase in pH is observed between 5to 6 ml. Then in the final analysis, increments of 0.2 ml of sodium hydroxide should be added, i.e., 5.0, 5.2, 5.4, 5.6, 5.8 and6.0).

4. After 6.0 ml, add 1 ml increments to the complex mixture and report the data.
5. Draw a graph between volume of sodium hydroxide added on x-axis and pH on the y-axis by using data obtained in the titration of glycine and complex solution.

Observations and calculations:

Data for analysis of complex of cupric-glycine by pH titration method

Glycine solution		**Preliminary study Complex solution**		**Final readings Complex solutio**	
Volume of sodiu hydroxide solution(ml)	**pH**	Volume of sodium hydroxide solution(ml)	**pH**	Volume ofsodiu hydroxide solution(ml)	**pH**
0		0		0	
1		1		1	
2		2		2	
3		3		3	
4		4		4	
5		5		5	
6		6		5.2	
		7		5.4	
		8		5.6	
				5.8	
				6.0	
				7.0	
				8.0	

Report: The complex stability constant (log β) of Copper - Glycine complex pH by titration method is----------. The donor acceptor ratio (n) of Copper - Glycine complex pH by titration method is---------- --.

Vision and Mission of the Institution

Vision

The East Point College of Pharmacy aspires to be a globally acclaimed institution, **recognized** for **excellence in** pharmaceutical education, research and nurturing students for **holistic development.**

Mission

M1 Create pharmacy graduates through **quality education**

M2 Promote innovation, **creativity**, and excellence **in teaching**, learning, and **research**

M3 **Inspire** integrity, teamwork, critical thinking**, personal** development, and ethics in **students** and lay **the** foundation for lifelong learning

M4 **Serve** the **healthcare, technological, scientific**, and **economic** needs of then **society**.