TECHNOCRATS

Lab Work Book of

Physical Pharmaceutics I

(BP- 306 P)

Department of Pharmacy

Lab Manual of

Physical Pharmaceutics I

(BP- 306 P)

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TECHNOCRATS
PUBLICATIONS

Lab Work Book
of

PHYSICAL PHARMACEUTICS I

(BP-306P)

(Strictly According to RGPV Syllabus)

Name : .....

Enrollment No. : .....

Institute : .....

Academic Session : .....

Department of Pharmacy

TECHNOCRATS
PUBLICATIONS

Vision of the Institute

To grow as an institute of Excellence for Pharmacy Education and Research and to serve the humanity by sowing the seeds of intellectual, cultural, ethical, and humane sensitivities in the students to develop a scientific temper, and to promote professional and technological expertise.

Mission of the Institute

M 1: To inculcate ethical, moral, cultural and professional values in students

M 2: To provide state of art infrastructure facilities to the staff and students so as to enable them to learn latest technological advancements

M 3: State of art learning of professionalism by the faculty and students

M 4: To produce well learned, devoted and proficient pharmacists

M 5: To make the students competent to meet the professional challenges of future

M 6: To develop entrepreneurship qualities and abilities in the students

PROGRAM OUTCOMES (POs)

1. **Pharmacy Knowledge:** Possess knowledge and comprehension of the core and basic knowledge associated with the profession of pharmacy, including biomedical sciences; pharmaceutical sciences; behavioral, social, and administrative pharmacy sciences; and manufacturing practices.
2. **Planning Abilities:** Demonstrate effective planning abilities including time management, resource management, delegation skills and organizational skills. Develop and implement plans and organize work to meet deadlines.
3. **Problem analysis:** Utilize the principles of scientific enquiry, thinking analytically, clearly and critically, while solving problems and making decisions during daily practice. Find, analyze, evaluate and apply information systematically and shall make defensible decisions.
4. **Modern tool usage:** Learn, select, and apply appropriate methods and procedures, resources, and modern pharmacy-related computing tools with an understanding of thelimitations.
5. **Leadership skills:** Understand and consider the human reaction to change, motivationissues, 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 well- being.
6. **Professional Identity:** Understand, analyze and communicate the value of their professional roles in society (e.g. health care professionals, promoters of health, educators, managers, employers, employees).
7. **Pharmaceutical Ethics:** Honour personal values and apply ethical principles in professional and social contexts. Demonstrate behavior 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.
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.
9. **The Pharmacist and society:** Apply reasoning informed by the contextual knowledge to assess societal, health, safety and legal issues and the consequent responsibilities relevant to the professional pharmacy practice.
10. **Environment and sustainability:** Understand the impact of the professional pharmacy solutions in societal and environmental contexts, and demonstrate the knowledge of, and need for sustainable development.
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-assess and use feedback effectively from others to identify learning needs and to satisfy these needs on an ongoing basis.

PEOs

PEO 1: To inculcate quality pharmacy education and training through innovative Teaching Learning Process.

PEO 2: To promote professionalism, team spirit, social and ethical commitment with effective interpersonal communication skills to boost leadership role assisting improvement in healthcare sector.

PEO 3: To enhance Industry-Institute-Interaction for industry oriented education and research, which will overcome healthcare problems of the society.

PEO 4: To adapt and implement best practices in the profession by enrichment of knowledge and skills in research and critical thinking

PEO 5: To generate potential knowledge pools with interpersonal and collaborative skills to identify, assess and formulate problems and execute the solution in closely related pharmaceutical industries and to nurture striving desire in students for higher education and career growth.

Course Outcomes (COs):

Student will be able to:

- CO1: Determine the physicochemical properties of pharmaceutical substances.
- CO2: Estimate the pH of the fluids.
- CO3: Estimate the pH of the liquids.
- CO4: Construct phase diagrams.
- CO5: Calculate phase diagrams.

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Experiment No. - 1

OBJECT:

To determine the solubility of drug at room temperature.

REFERENCES:

Gaud R S & Gupta G D , "Practical Physical Pharmacy", 1st edition , Reprint 2008, CBS Publishers & Distributors, New Delhi, Page No-112-113

REQUIREMENT:

Benzoic acid, NaOH, Hcl, Distilled water, Thermometer, Beaker

THEORY:

Solubility is a chemical property referring to the ability for a given substance, the solute, to dissolve in a solvent. It is measured in terms of the maximum amount of solute dissolved in a solvent at equilibrium. The resulting solution is called a saturated solution.

Solubility is the property of a solid, liquid, or gaseous chemical substance called solute to dissolve in a solid, liquid, or gaseous solvent. The solubility of a substance fundamentally depends on the physical and chemical properties of the solute and solvent as well as on temperature, pressure and the pH of the solution. The extent of the solubility of a substance in a specific solvent is measured as the saturation concentration, where adding more solute does not increase the concentration of the solution and begins to precipitate the excess amount of solut**e.**

PROCEDURE:

1. Thoroughly clean all glassware using detergent and chromic acid solution
2. Rinse the glasswares two to three times using distilled water and dry completely in the oven.
3. Take 25 ml of distilled water in a beaker and heat slightly at more than room temperature and add excess amount of benzoic acid in the beaker with constant stirring
4. Cool this beaker at room temperature
5. Take 5 ml of supernatant liquid from the saturated solution at room temperature and transfer in a conical flask
6. Titrate this solution using standardized 0.1N NaOH and phenopthelin as an indicator

7. Repeat step 5 and 6 at least three times
8. In another flask take 25 ml of distilled water in a beaker and cool it at 10.c
9. Add excess amount of benzoic acid and repeat step 3 to 6
10. All data's presented in the form of table.

OBSERVATION:

S. No.	Temperature	Burette Initial reading (a)	Burette final reading (b)	Volume of 0.1 N NaOH required to neutralization (b-a) ml
1	Room temperature			
2				
3				
4				

CALCULATION:

Calculation of solubility of benzoic acid at room temperature

$$\mathbf{N_1V_1 = N_2V_2}$$

solution alkali

where, N_1 is normality of solution, V_1 is the volume of solution withdrawn at room temperature for titration, N_2 is the normality of NaOH and V_2 is the volume required of NaOH required to neutralize the withdrawn solution.

$N_1 = N_2V_2/V_1$

Solubility of Benzoic acid at room temperature is determined by the following formula solubility of benzoic acid per 100 ml of solution (M)

$$M = \frac{N_1 E \times 100}{1000} g/100ml$$

.....

.....

.....

.....

RESULT:

Solubility of benzoic acid at room temperature is .....%

VIVA QUESTION:

Q.1. What is solubility?

Ans- .....
.....
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.....

Q.2. Define solute and solvent?

Ans- .....
.....
.....
.....

Q.3. Define titration?

Ans- .....
.....
.....
.....

Q.4. What is saturated solution?

Ans- .....
.....
.....
.....

Q.5. Do Solubility depends upon temperature. Explain

Ans- .....
.....
.....
.....

Experiment No. - 2

OBJECT:

To determine the pka value by Henderson HassleBalch equation.

REFERENCE:

CVS Subrahmanyam, "Laboratory Manual of Physical Pharmaceutics", First ed, 2002 vallabh prakashan, Page no 92

THEORY:

A dissociation constant is a specific type of equilibrium constant that measures the propensity of a larger object to separate (dissociate) reversibly into smaller components, as when a complex falls apart into its component molecules, or when a salt splits up into its component ions.

The Henderson–Hasselbalch equation describes the derivation of pH as a measure of acidity (using pKa, the negative log of the acid dissociation constant) in biological and chemical systems. The equation is also useful for estimating the pH of a buffer solution and finding the equilibrium pH in acid-base reactions (it is widely used to calculate the isoelectric point of proteins).

The equation is given by:

$$pH = pka + \log \frac{\text{Ionized acid}}{\text{Unionized acid}}$$

MATERIAL AND APPARATUS:

Salicylic acid standard sodium hydroxide solution ph meter conical flask pipettes and indicator

PROCEDURE:

1. 10 ml 0f 0.5 5 w/v solution of salicylic acid in methanol is pipetted out to a conical flask.
2. This is then titrated against 0.5 N sodium hydroxide solution using methyl red as indicator to complete neutralization. The burette reading is noted.
3. Similarly 10 ml of 0.5% w/v salicylic acid solution is taken into a flask and titrated against standard 0.5 N sodium hydroxide solutions to 50 % neutralization point.
4. The ph of this half neutralized solution is recordedusing a ph meter.

OBSERVATION AND CALCULATION:

Let the volume of 0.5 N sodium hydroxide consumed in titration with 10 ml o.5% salicylic acid solution = x ml

Volume of 0.5 N sodium hydroxide solution required for half neutralization = x/2 ml

pHof half neutralization solution =……..

RESULT:

……

VIVA QUESTION:

Q.1. What are the significances of pka value?

Ans- .....

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Q.2. What is the relation between pka and pkb?

Ans- .....

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Q.3. What is the unit of pka?

Ans- .....

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Q.4. What is the importance of pka in pharmacy?

Ans- .....

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Q.5. What are the other methods of determination of pka

Ans- .....

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Experiment No. - 3

OBJECT:

To Determine the Partition co- efficient of benzoic acid in benzene and distilled water.

REFERENCE:

Gaud R S & Gupta G D , "Practical Physical Pharmacy", 1st edition , Reprint 2008, CBS Publishers & Distributors, New Delhi, Page No.- 148-151.

REQUIREMENTS:

A)Chemicals/Reagents: Benzene, Benzoic acid, 0.05M sodium hydroxide , Distilled water.

B) Equipments/glassware's: Separating funnel, conical flask, Burette, Pipette, Reagent bottles

THEORY:

If the insufficient amount of substance is added in two immiscible liquids to the saturation, it gets distributed in two layers at a definite ratio, this phenomenon is called as distribution law or partition law. The ratio constant is called as partition coefficient or distribution coefficient. It is independent of the total amount of the substance dissolved.

It was observed that if the solute has equal molecular weight in both solvents then the ratio of the concentration of the solute in the two immiscible solvent is found to be constant

$$k = c_1 / c_2$$

Where, K is a constant kown as distribution or partition coefficient.C_1 & C_2 are the concentration can be represented in g/litre or gram equivalent/litre.

PROCEDURE:

1. Take five reagent bottles and clean these bottles by reagent and rinse through the distilled water.
2. Prepare composition of the solution as given in the table A.
3. Transfer these solutions in clean and dry reagent bottles and place the level 1, 2,3,4,5 respectively.
4. Place the stopper on each bottle & shake it for 30 min.

5. Variability of the result depends on the shaking hence more and effective shaking is essential for reproducible results.
6. Now take this mixture in separating funnel and keep aside for about to 30 min.
7. Separate carbon tetrachloride and aqueous layer in two conical flasks.
8. Intermediate liquid can not be collected as it contains little of both liquids.
9. Put the label on both conical flasks with the samples taken originally.
10. Pipette out 10ml of the aqueous layer and transfer in another conical flask.
11. Separate two layer by separating funnel.
12. Withdraw 10 ml sample of organic layer and in conical flask.
13. Add 2 to 3 Drops of phenolphthalein as indicator and titrate it using 0.05M NaoH
14. Repeat the procedure at least three times for accuracy and confirm reproducible.
15. Record all estimation values carefully.

OBSERVATION:

TABLE A: PREPARATION OF SOLUTION:-

S. No.	container	composition
1.	1	1.0 g of benzoic acid + 50ml of benzene + 50ml of water
2.	2	1.5 g of benzoic acid + 50 ml of benzene +50 ml of water
3.	3	2.0 g of benzoic acid + 50 ml of benzene + 50 ml of water
4.	4	3.0 g of benzoic acid +50 ml of benzene + 50 ml of water
5.	5	4.0 g of benzoic acid+ 50 ml of benzene + 50 ml of water.

TABLE B: TITRATION OF AQUEOUS LAYER

S. No.	Container	Volume taken (ml)	Initial reading (Burette reading)	Final reading (Burette reading)	Volume used of 0.05 M Sodium hydroxide (ml)
1.	A_1				V_1
2.	B_2				V_2
3.	C_3				V_3
4.	D_4				V_4
5.	E_5				V_5

TABLE C: TITRATION OF ORGANIC LAYER

S. No.	Container	Volume taken (ml)	Initial reading (Burette reading)	Final reading (Burette reading)	Volume used of 0.05 M Sodium hydroxide (ml)
1.	A_1'				V_1'
2.	B_2'				V_2'
3.	C_3'				V_3'
4.	D_4'				V_4'
5.	E_5'				V_5'

Calculation: Calculate the concentration of benzoic acid in benzene (organic layer) C_{org} and in distilled water(aqueous layer) C_{aqs}.

S. NO	Container	Concentration in organic layer (C_{org})	Concentration in aqueous layer (Caqs)	Partition coefficient k (Corg/Caqs)	Average reading of C_{org}/C_{aqs}
1.	A_1/A1'				
2.	B_2/ B_2'				
3.	C_3/ C_3'				
4.	D_4/ D_4'				
5.	E_5/ E_5'				

RESULT:

The partition coefficient of benzoic acid in benzene & distilled water =

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Plot the graph between log Caqs against log $C_{org,}$ and determine the value of n and k by straight line.

VIVA QUESTION:

Q.1. What is the distribution coefficient?

Ans- .....

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Q.2. How to determine distribution coefficient?

Ans- .....

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Q.3. What is the role of partition coefficient in the dosage formulation?

Ans- .....

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Q.4. Give the unit of partition coefficient?

Ans- .....

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Q.5. What is the role of partition coefficient in the absorption?

Ans- .....

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Experiment No. - 4

OBJECT:

To Determine the Partition co- efficient of Iodine between CCl4 and distilled water.

REFERENCE:

Gaud R S & Gupta G D , "Practical Physical Pharmacy", 1st edition , Reprint 2008, CBS Publishers & Distributors, New Delhi, Page No.- 142-144.

REQUIREMENTS:

a)Chemicals/Reagents:- Carbon tetrachloride, iodine, distilled watet

b) Equipments/glassware's:- Separating funnel, conical flask, Burette, Pipette, Reagent bottles

THEORY:

When an excess amount of substance is added in two immiscible liquids to the saturation, it gets distributed in two layer at a definite ratio, this phenomenon is called as distribution law or partition law. The ratio constant is called as partition coefficient or distribution coefficient. It is independent of the total amount of the substance dissolved.

It was observed that if the solute has equal molecular weight in both solvents then the ratio of the concentration of the solute in the two immiscible solvent is found to be constant

$$k = c_1 / c_2$$

Where, K is a constant known as distribution or partition coefficient.C_1 & C_2 are the concentration of solute in two immiscible liquid, represented in g/litre or gram equivalent/litre.

PROCEDURE:-

1. Prepared a saturated solution of iodine in carbon tetrachloride(stock solution).
2. Take three reagent bottles and clean these bottles by reagent and rinse through the distilled water.
3. Prepare composition of the solution as given in the table.
4. Transfer these solution in clean and dry reagent bottles and be it as A,B, and C respectively.
5. Place the stopper on each bottle & shake it for 30 min. or shake using wrist action shaker and rotatary shaker.

6. Variability of the result depends on the shaking hence more and effective shaking is essential for reproducible results.
7. Now take this mixture in separating funnel and keep aside for about to 30 min.
8. Separate carbon tetrachloride and aqueous layer in two conical flasks.
9. Intermediate liquid can not be collected as it contains little of both liquids.
10. Put the label on both conical flasks with the samples taken originally.
11. Pipette out 10ml of the aqueous layer and transfer in another conical flask.
12. Add 2 to 3 drops of starch solution & titrate it against 0.01 sodium thiosulphate solution.
13. Record the titration value and repeat the steps 10 and 11.
14. Similarly, titrate the aqueous layer from other containers.
15. Pipette 10 ml of carbon tetrachloride layer in a dry and clean conical flask.
16. Add starch solution 2 to 3 drops as an indicator and estimate concentration of iodine by titration with 0.01 N sodium thiosulphate solution.
17. Repeat the step 15 and 16 till you get constant burette reading.
18. Similarly, titrate other carbon tetrachloride solution as step 15 to 17.
19. Take all readings carefully.
20. Calculate concentration of iodine in both phases , that is aqueous and organic phase.

TABLE- A:PREPARATION OF SOLUTION.

S. No.	Container	Composition
1.	A	25 ml stock solution +100 ml of distilled water
2.	B	15 ml stock solution + 10 ml pure CCl_4 +100 ml of distilled water
3.	C	5 ml stock solution + 20 ml pure CCl_4 +100 ml of distilled water

OBSERVATION- ‘

TABLE B: TITRATION OF AQUEOUS LAYER

S. No.	Container	Volume taken (ml)	Initial reading (B u r e t t e reading)	Final reading (B u r e t t e reading)	V o l u m e used of 0.01 M Sodium Thiosulphate (ml)
1.	A				V_1
2.	B				V_2
3.	C				V_3

TABLE C: TITRATION OF ORGANIC LAYER

S. No.	Container	Volume taken (ml)	Initial reading (B u r e t t e reading)	Final reading (B u r e t t e reading)	V o l u m e used of 0.01 M Sodium Thiosulphate (ml)
1.	A’				V_1'
2.	B’				V_2'
3.	C’				V_3'

CALCULATION:-

FOR AQUEOUS LAYER-

Concentration of iodine in container A

$N_1V_1 = N_2V_2$

$N_1 = 0.01 * V_2/10$

Concentration of iodine in water layer (C_1)

$C_1 = (0.01 * V_2 * 127)/10$ mole /litre

Similarly calculate the concentration of iodine in other flask(B& C).

FOR ORGANIC LAYER-

Concentration of iodine in container A’

$N_1 `V_1 ` = N_2 `V_2 `$

$N_1 ` = 0.01 * V_2 `/10$

Concentration of iodine in water layer(C_2)

$C_2 = (0.01 * V_2 ` * 127)/10$ mole /litre

Similarly calculate the concentration of iodine in other flask (B` & C`).

Partition coefficient $K = C_1/C_2$

RESULT:

The partition coefficient of iodine between carbon tetrachloride & distilled water

=_____.

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VIVA QUESTION:

Q.1. What is the distribution coefficient?

Ans- .....

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Q.2. How to determine distribution coefficient ?

Ans- .....

.....

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.....

Q.3. What is the role of partition coefficient in the dosage formulation?

Ans- .....

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Q.4. Give the unit of partition coefficient?

Ans- .....

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Q.5. What is the role of partition coefficient in the absorption?

Ans- .....

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Experiment No 5

OBJECT:

To determine the Critical Solution temperature (CST) for the phenol-water system, and to study the effect of added impurity (NaCl) on the CST.

REFERENCES:

CVS Subrahmanyam and S.G. Vasanthraju, "Laboratory Manual Of Physical Pharmacy-I", First ed, 1999, Vallabh Prakashan, Page no 46.

THEORY:

The number of homogeneous, mechanically separable and physically distinct parts of a heterogeneous system is known as the number of phases, P, of the system.

Each phase is separated from other phases by a physical boundary.

When equilibrium exists between a number of phases under external controlling conditions such as temperature, pressure, and concentration, the following relationship holds good:

$$P + F = C + 2$$

where P = number of Phases in equilibrium,

C = number of Components in the system, and

F = number of degrees of Freedom.

Equation is called the Phase Rule, which relates the phases, components and degrees of freedom of the system.

When 2 partially miscible liquids are mixed and shaken together, we get 2 solutions of different compositions. For example, on shaking phenol and water, we get 2 layers: the upper layer is a solution of water in phenol, and the lower layer is a solution of phenol in water. At a fixed temperature, the composition of each solution is fixed, and both the solutions are in equilibrium.

Two solutions of different compositions existing in equilibrium with one another are known as conjugate solutions. Above a particular temperature, such solutions are completely miscible in all proportions. Such a temperature is known as the Critical Solution Temperature (CST) or Consolute Temperature. As the mutual solubility increases with temperature in this particular case, it is known as Upper Consolute Temperature.

The CST is markedly affected by pressure and also by the presence of impurities. Hence, the CST may

be taken as a criterion for the purity of a substance. The solubility changes in the CST for Phenol - Water system is affected by adding an electrolyte to it on miscibility temperature.

Addition of sodium chloride to the phenol – water system enhances its upper consolute temperature. The reasons as follows so chloride is more soluble in water compared to its solubility in phenol.

MATERIALS REQUIRED:

Hard Glass Test tubes (or boiling tube as air jacket),transition temperature apparatus, thermometer (graduated to 0.1ºC), stirrer, beakers (500ml, 100 ml), volumetric flask (100 ml), pipette (1 ml, 2 ml), phenol, distilled water, sodium chloride, hot plate.

PROCEDURE:

Stock solution, phenol 80%

Prepare 80% v/v solution of phenol in water by taking 80 ml of phenol and 20 ml of water. At this proportion, water and phenol are completely miscible. This stock solution is used for the preparation of different concentration of phenol in water.

Stock solution, sodium chloride 1% w/v

Accurately weigh one gram of sodium chloride and transfer into a 100 ml volumetric flask. Add distilled water and dissolve the salt. Finally make up the volume to 100 ml mark.

METHOD:

1. Prepare various concentrations of sodium chloride (0.2, 0.4, 0.6, 0.8% w/v) in water (10ml) using stock solution.
2. Take 2.5 ml of phenol and 2.5 ml of water into the transition temperature tube. Determine the miscibility temperature. Note the temperature at which the opalescence disappears.
3. Remove transition temperature tube from the water bath and stir to allow cooling. Note the temperature at which the opalescence reappears.
4. Determine the mean of t1 and t2 and record. This data represent the zero concentration of sodium chloride solution.
5. Similarly determine miscibility temperature of phenol sodium chloride solutions for various other concentration of sodium chloride by following step 3 and 4.
6. Plot a graph of miscibility temperature versus % concentration of sodium chloride
7. determine miscibility temperature of the unknown solution.

8. Estimate the %composition of sodium chloride in the unknown solution from the standard plot

Sr. No.	Percentage of NaCl w/v	Miscibility temperature, °c		
		t1	t2	mean
1	0.0			
2	0.2			
3	0.4			
4	0.6			
5	0.8			
6	1.0			

RESULTS

CST of unknown sample =

The percentage composition of sodium chloride in the given solution =

VIVA QUESTION:

Q.1. What is CST?

Ans- .....
.....
.....
.....

Q.2. What is stock solution?

Ans- .....
.....
.....
.....

Q.3. What is conjugate solution?

Ans- .....
.....
.....
.....

Q.4. Write importance of CST in pharmacy?

Ans- .....
.....
.....
.....

Q.5. What is upper consolute temperature ?

Ans- .....
.....
.....
.....

Experiment No. - 6

OBJECT:

To determine surface tension of given liquids by drop count and drop weight method.

REFERENCES:

Gaud R.S.& Gupta G.D. "Practical Physical Pharmacy," CBS Publishers & distributors, New Delhi, First Edition 2001, Page no.87

REQUIREMENTS:

Stalagmometer, specific gravity bottle, distilled water, experimental liquid.

THEORY:

The attractive force exerted upon the surface molecules of a liquid by the olecules beneath that tends to draw the surface molecules into the bulk of the liquid and kes the liquid assume the shape having the least surface area In the drop number method, the number of drops formed by equal volumes of two liquid is counted.

If m1 and m2 is the mass of one drop of each of the liquid having densities d1 and d2 respectively. If n1 and n2 is the number of drops formed by volume v of the two liquids, then their surface tensions are related as

$$\frac{¥1}{¥2} = \left(\frac{d1}{d2}\right) \times \left(\frac{n2}{n1}\right)$$

One of the liquid is water its surface tension and density are known. Then their surface tension of the given liquid can be calculated.

PROCEDURE:

1. Clean the stalagmometer with chromic acid then wash with water and dry it.
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.

4. Clamp the stalagmometer 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 the exercise to take three to four readings.
6. Rinse the stalagmometer with alcohol and dry it
7. Suck the given liquid in the stalagmometer and count the drops formed between two points.
8. Take a clean dry weighing bottle also weighs this volume of water as well as sample liquid.
9. Put the values in formula and calculate the surface tension.

OBSERVATIONS:

Room temp= t_oC = ……..

Density of water= d_{w} =……

Surface tension of water= ¥ dynes/cm = ……

No of drops From a Fixed Volume				Mean
Liquid	1…..	2…..	3….	n_l =
Water	1….	2….	3….	n_w =

Weight of drop				Mean
Liquid	1…..	2…..	3….	n_l =
Water	1….	2….	3….	n_w =

Weight of empty specific gravity bottle=W_1gram =……..

Weight of specific gravity bottle+water=W_2 gram =…….

Weight of empty sp.gravity bottle+liquid=$w_3$gram=……..

Weight of water= (W_2-W_1) gram =……..

Weight of liquid= (W_3-W_1) gram=……..

CALCULATIONS:

DENSITY OF THE LIQUID

$$D_l = \frac{(W3 - W1)}{(W2 - W1)} \times dw$$

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SURFACE TENSION OF LIQUID=

$$\frac{¥1}{¥2} = \left(\frac{d1}{d2}\right) \times \left(\frac{n2}{n1}\right)$$

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.....

RESULT:

The surface tension of liquid is found to be ……dynes/cm.

VIVA QUESTIONS

Q.1 What is surface tension?

Ans- .....
.....
.....
.....

Q,2 What is density?

Ans- .....
.....
.....
.....

Q.3 What are the factors affecting surface tension?

Ans- .....
.....
.....
.....

Q.4 Write formula of surface tension.

Ans- .....
.....
.....
.....

Q.5. Give name of 5 surfactants?

Ans- .....
.....
.....
.....

Experiment No. - 7

OBJECT:

To determine HLB number of a surfactant by saponification method.

REFERENCES:

Subramanyam C.V.S, "Text book of physical pharmaceutics", vallabh prakashan second reprint edition 2002, page no: 150.

REQUIREMENTS:

Stearic acid, KOH, alcohol.

THEORY:

HLB (Hydrophile-Lipophile Balance) is an empirical expression for the relationship of the hydrophilic ("water-loving") and hydrophobic ("water-hating") groups of a surfactant. The table below lists HLB values along with typical performance properties. The higher the HLB value, the more water-soluble the surfactant.
The HLB system is particularly useful to identify surfactants for oil and water emulsification.

Surfactants are typically ampiphilic molecules that contain both hydrophilic and lipophilic groups. The hydrophile-lipophile balance (HLB) number is used as a measure of the ratio of these groups. It is a value between 0-60 defining the affinity of a surfactant for water or oil.

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HLB is that emulsifier having low HLB value tend to be oil soluble and materials having high values tend to be water sol uble.

$$HLB = 20\left(1 - \frac{S}{A}\right)$$

Where,

S= saponification number of the ester

A= acid number of the acid.

Application of Surfactant on HLB value

HLB value	Application
4-6	o/w emulsifiers
7-9	Wetting agent
8-18	o/w emulsifiers
13-15	Detergent
10-18	Solubilizer

PROCEDURE:W

1. Preparation of 0.5N alcoholic KOH:

(i) gm of KOH was dissolved in 5 ml distilled water in volumetric flask and total volume was made upto 100ml with alcohol.

(ii) Allowed it to stand for 24 hours & the clear liquid was separated by decantation (Clear solution was used)

2. Determination of saponification number:

a. Glyceryl monostreate was weighed 0.5 gm and transferred to round bottom flask. 15 ml of alcoholic potassium hydroxide was added to it and refluxed on boiling water bath for 1 hour.

b. Alcoholic potassium hydroxide (Without glycerol monostearate) 15 ml was refluxed separately on boiling water bath for half an hour as blank reading.

c. Both the solutions were cooled at room temperature and were separately titrated against 0.5 N hydrochloric acid using phenolphthalein as the indicator (End point- pink to colorless or slightly yellowish).

d. Let the reading for sample be V1 and blank be as V2

3. Determination of acid number:

a. Stearic acid 0.5 gm was added to mixture of 10 ml of alcohol and 10 ml ether.

b. Solution of stearic acid was titrated against 0.1N solution hydroxide using phenolphthnalein as an indicator, it the titre reading be V3.

4. Calculation of HLB value:

Saponification number(S) and acid number (A) was calculated and HLB value was determined using formula,

HLB=20(1-S/A)

OBSERVATION:

S.No.	Parameter	Value
1.	Saponification number	
2.	acid number:	
3.	HLB	

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RESULT:

The HLB value was fond to be……

VIVA QUESTIONS

Q.1 What is HLB value

Ans- .....

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Q.2 Define surfactant.

Ans- .....

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Q.3 What is saponification

Ans- .....

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Q.4 What is acid number.

Ans- .....

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Q.5 What is drawback of HLB scale?

Ans- .....

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Experiment No. 8

OBJECT:

To study the adsorption behavior of acetic acid on activated charcoal.

REFERENCE:

Subrahmanyam CVS and Vasantharaju S. G, Laboratory manual of Physical Pharmacy. First edition-2002, Vallabh Prakashan Delhi, Page no-96.

APPARATUS AND CHEMICAL REQUIREMENT:

Activated char coal, Acetic acid. Phenolphthalein indicator, NaOH Conical flask, Burette, Pipette, Reagent bottles

THEORY :

Adsorption is the adhesion of atoms, ions or molecules from a gas, liquid, or dissolved solid to a surface. This process creates a film of the adsorbate on the surface of the adsorbent. This process differs from absorption, in which a fluid is dissolved by or permeates a liquid or solid (the absorbent), respectively. Adsorption is a surface-based process while absorption involves the whole volume of the material. The term sorption encompasses both processes, while desorption is the reverse of it. Adsorption is a surface phenomenon.

Similar to surface tension, adsorption is a consequence of surface energy. In a bulk material, all the bonding requirements (be they ionic, covalent, or metallic) of the constituent atoms of the material are filled by other atoms in the material. However, atoms on the surface of the adsorbent are not wholly surrounded by other adsorbent atoms and therefore can attract adsorbates. The exact nature of the bonding depends on the details of the species involved, but the adsorption process is generally classified as physisorption (characteristic of weak van der Waals forces) or chemisorption (characteristic of covalent bonding). It may also occur due to electrostatic attraction.

Activated carbon, also called activated charcoal, is a form of carbon processed to have small, low-volume pores that increase the surface area available for adsorption or chemical reactions. Activated is sometimes substituted with active.

PROCEDURE:

1. Weigh about 2.0 gm of activated charcoal and note the exact weight. Prepare six packets.

2. Transfer the activated charcoal samples carefully into the acetic acid solution bottles.

3. Cork the bottles securely.

4. keep the bottle in a constant mild temperature water bath and shake it for half hour.

5. After equilibrium is attained, remove the bottles from the constant temperature water bath.

6. Filter the acetic acid solution using whatman paper no.01.

7. Pipette out 10 ml of filtrate into two conical flasks.

8. Titrate them against 0.05 N sodium hydroxide solution using phenolphthalein indicator.

9. Determine the equilibrium concentration and other parameters as indicated in table.

10. Draw a figure by taking Log (x/m) + 2 on y axis and Log C+2 on x axis.

OBSERVATIONS

Y intercept = Log K =

Slope = (1/n) =

K = antilog =

N = (1/slope) =

$C = N_1V_1/V_2$

N_1 = Normality of Na0H

V_1 = Volume of Na0H

V_2 = Volume of acetic acid

C = Normality of acetic acid remain unabsorbed

X = (Co – C) M

X = Amount of acetic acid absorbed

Co = Initial concentration of acetic acid

C = Eq. concentration of acetic acid

M = Mol. weight of acetic acid

m = Weight of charcoal

Table A : Preparation of different concentration of acetic acid

S. No.	Acetic acid stock solution, ml	Distilled water, ml	Concentration of acetic acid, N
A	5	95	0.05
B	10	90	0.1
C	20	80	0.2
D	30	70	0.3
E	40	60	0.4
F	50	50	0.5

Table B : Data generated for the adsorption studies for acetic acid on charcoal

Flask No.	*Volume of NaOH consumed, ml*		*Volume of NaOH consumed, ml (Mean ml)*	*Equillibrium conc. Of acetic acid C**	*Log C+2*	*X #*	*x/m*	*Log (x/m) + 2*
	Initial Ml	Final ml						
A 1								
2								
B 1								
2								
C 1								
2								
D 1								
2								
E 1								
2								
F 1								
2								

RESULT AND DISCUSSION:

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VIVA QUESTIONS

Q-1. What is adsorption ?

Ans- .....

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Q-2. What is adsorbent ?

Ans- .....

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Q-3. What is adsorbate ?

Ans- .....

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Q-4. Describe the application of adsorption in pharmacy.

Ans- .....

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Q-5. Describe the factor influencing the adsorption of drugs.

Ans- .....

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Experiment No. 9

OBJECT:

To determine of critical micellar concentration of surfactants.

REFERENCES:

Subramanyam C.V.S, "Text book of physical pharmaceutics", vallabh prakashan second reprint edition 2002, page no: 327.

REQUIREMENTS:

sodium lauryl sulfate, stalagmometer

THEORY:

In colloidal and surface chemistry, the critical micelle concentration (CMC) is defined as the concentration of surfactants above which micelles form and all additional surfactants added to the system go to micelles.

The CMC is an important characteristic of a surfactant. Before reaching the CMC, the surface tension changes strongly with the concentration of the surfactant. After reaching the CMC, the surface tension remains relatively constant or changes with a lower slope. The value of the CMC for a given dispersant in a given medium depends on temperature, pressure, and (sometimes strongly) on the presence and concentration of other surface active substances and electrolytes.

PROCEDURE:

1. Stock solution 250ml was prepared by dissolving 250mg sodium lauryl sulfate in distilled water (1mg/ml).
2. solution of different strengthen was prepared in distilled water with 5,10,15,20,25,30,35,40mg of sodium lauryl sulfate per 50ml of distilled water, from the stock solution.
3. Surface tension of all the solutions of sodium lauryl sulfate was determined using stalagmometer.
4. Graph of surface tension v/s concentration of sodium lauryl sulfate was plotted.
5. Critical micelle concentration of a given surfactant was determined using formula CMC=20C/1000M

where, M is Molecular weight of the surfactant and C is CMC in mg/ml.

OBSERVATION:

Sr. No.	Concentration(SLS in mg)	Surface tension
1	5	
2	10	
3	15	
4	20	
5	25	
6	30	
7	35	
8	40	

RESULT:

Critical micellar concentration of surfactants was found to be ……

VIVA QUESTIONS

Q.1 What is CMC?

Ans- .....

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Q.2 What is stock solution?

Ans- .....

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Q.3 What is surface active agents?

Ans- .....

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Q.4 What is micelle?

Ans- .....

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Q.5 What is micelle solubilization.

Ans- .....

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Experiment No. 10

OBJECT:

Determination of stability constant of glycine –copper complex by pH titration method.

REFERENCE:

Mohanta Guru Prasad & Manna Prabal Kumar, "Physical Pharmacy Practical Text", 1st Edition 2006, Pharma Book Syndicate,Hyderabad, Page no.- 80-84

APPARATUS & MATERIALS REQUIRED:

Glycine hydrochloride, cupric chloride, 0.259 N sodium hydroxide, pH meter , burette, beakers, volumetric flasks etc.

THEORY:

Glycine & Copper complex is a chelate type metal ion complex and the chelation of cupric ion by glycine is represented by :

$Cu^{+2} + 2NH_3^+CH_2COO^- = Cu\ (NH_2CH_2COO)_2 + 2H^+$

In glycine copper complex, copper ion is the metal (M) & the glycine is the ligand (A).

The equation would be:

M+A = MA; $K = [MA]/[M][A]$

MA+A = MA_2; $K = [MA_2]/[MA][A]$

M + 2A = MA_2 ; and $\beta = k_1k_2 =$ $K = [MA_2]/[MA][A]^2$

Where k_1 and k_2 are formation constant & β is the equilibrium constant for overall reaction (also known as stability constant).

The average number of ligand groups bound per metal ion is expressed as:-

$$\text{Average no.}(\bar{n}) = \frac{\text{[Total concentration of ligand bound]}}{\text{[Total concentration of metal ion]}}$$

The concentration of free glycine [A] in this Complexation reaction can be obtained as:-

P[A] = ½ log β at $\bar{n} = 1$;

P[A] = log k_1 at $\bar{n} = 1/2$;

P[A] = log k_2 at $\bar{n} = 3/2$;

PROCEDURE:

1. Titration of Glycine Hydrochloride with Sodium hydroxide:

- 100 ml glycine hydrochloride solution at $3.34*10^{-2}$ mole per litre concentration is prepared. (molecular weight of glycine hydrochloride is 111.53.0.372 g of glycine hydrochloride is required to be dissolved & volume is to be made 100ml).
- 75 ml glycine hydrochloride solution is pipetted out & taken in a conical flask. The p^H of the solution is measured.
- 0.259 N sodium hydroxide is added gradually and p^H of the mixture is measured after each 1ml addition of sodium hydroxide.

2. Titration of Glycine hydrochloride and Copper Complex with sodium hydroxide:-

- 100 ml solution containing $3.34* 10^{-2}$ mole per litre of glycine hydrochloride and $9.45 * 10^{-3}$ mole per litre of cupric chloride is prepared.(The molecular weight of cupric chloride is 134.45.0.127 g of cupric chloride is required).
- 75 ml of glycine hydrochloride and cupric chloride solution is pipetted out and taken in a conical flask. The p^H of the solution is measured.

0.259 N sodium hydroxide is added gradually and p^H of the mixture is measured after each 1 ml addition of sodium hydroxide.

3. A graph is drawn taking volume of sodium hydroxide in X- axis & p^H in Y-axis

4. The value of $\bar{n}$ and p[A] at different p^H are computed from the described equation.

5. The formation curve is plotted using p[A] in X-axis and $\bar{n}$ in Y-axis.

6. The value of log K_1 at $\bar{n}$ = ½ log K_2 at $\bar{n}$ =3/2 and

½ log β at $\bar{n}$ =1 is obtained by extrapolation of formation curve. The value of log K_1, log K_2 and ½ log β are equal to corresponding p[A].

OBSERVATION & CALCULATION:

TABLE NO. 1

Room Temperature _____ ̊C

S.No	Volume of 0.259 N NaoH added	P^H of glycine hydrochloride mixture with sodium hydroxide	P^H of glycine hydrochloride and copper complex mixture with sodium hydroxide
1.	0		
2.	1		
3.	2		
4.	3		
5.	4		
6.	5		
7.	6		
8.	7		
9.	8		

TABLE NO 2.

P^H	ml of NaoH Solution per 75ml sample	Total legand bound concentration	$\bar{n}$	P[A]
3.5				
4.0				
4.5				
5.0				
5.5				
6.0				
6.5				
7.0				
7.5				
8.0				

RESULT :

The stability constant of copper- glycine complex is _____ at _____ ̊C

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(**Note:** The log β value for this complex is around 15.3**).**

VIVA QUESTIONS

Q.1. What are the importance of Complexation ?

Ans- .....
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Q.2. What is the unit of stability constant ?

Ans- .....
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Q.3. What are the other methods of determination of stability constant ?

Ans- .....
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Q.4. Give types of complexation

Ans- .....
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Q.5. Define complexation

Ans- .....
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