

University of Kerbala  
College of Pharmacy  
Department of Pharmaceutics



# LABORATORY MANUAL for PHYSICAL PHARMACY II

*Second Year Students*

By

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# C

## ONTENTS

Partition Coefficient.....	3
Complexation.....	7
Chemical Kinetics.....	11
Surfactants.....	15
Solubilization by Surfactants .....	20
Viscosity .....	23

## Lab. 1

# PARTITION COEFFICIENT

### Introduction

If a liquid or solid substance is added to a mixture of two immiscible liquids, it will become distributed between the two layers in a definite concentration ratio.

If  $C_1$  and  $C_2$  are the substance equilibrium concentrations in solvent 1 and solvent 2, respectively, the equilibrium expression becomes:

$$C_1 / C_2 = K$$

The equilibrium constant,  $K$ , is known as the distribution ratio, distribution coefficient, or partition coefficient.

In this experiment, the partition coefficient of iodine between water and chloroform will be determined.



### Materials and equipment

- Iodine 1% (in  $\text{CHCl}_3$ ), potassium iodide 10%, distilled water, and sodium thiosulfate 0.1 N and 0.02 N
- Separatory funnel, burette, conical flasks, and graduated cylinder.

### Procedure

1. In dry stoppered separatory funnel, put 20 ml of 1 % iodine in chloroform.
2. Add 50 ml distilled water to the funnel.
3. Shake the flask for 15 min until equilibrium is established, allow to stand for 15 min until the phases is completely separated.
4. Separate the organic layer form the aqueous layer.
5. Withdraw 10 ml of the aqueous layer and titrate against 0.02 N sodium thiosulfate until the light brownish color disappear.
6. Withdraw 5 ml of the organic layer, add 5 ml of 10 % potassium iodide with vigorous shaking, and then titrate against 0.1 N sodium thiosulfate until the light brownish color disappear.

**Calculations:**

Iodine is distributed between the aqueous phase and the chloroform phase.

**Aqueous phase:**

The (mls) of sodium thiosulfate consumed in the titration is equivalent to the amount of iodine present.



$$\begin{array}{ccc} \text{N}_1 \times \text{V}_1 & = & \text{N}_2 \times \text{V}_2 \\ \text{I}_2 & & \text{Na}_2\text{S}_2\text{O}_3 \end{array}$$

$$\text{N}_1 \times 10 = 0.02 \times \text{E.P}$$

$\text{N}_1$  = concentration of iodine in the aqueous layer

**Organic phase:**

The (mls) of sodium thiosulfate consumed in the titration is equivalent to the amount of iodine present.

$$\begin{array}{ccc} \text{N}_1 \times \text{V}_1 & = & \text{N}_2 \times \text{V}_2 \\ \text{I}_2 & & \text{Na}_2\text{S}_2\text{O}_3 \end{array}$$

$$\text{N}_1 \times 5 = 0.1 \times \text{E.P}$$

$\text{N}_1$  = concentration of iodine in the organic layer

$$\text{Partition Coefficient (K)} = \text{N}(\text{I}_2 \text{ H}_2\text{O}) / \text{N}(\text{I}_2 \text{ CHCl}_3)$$

Group:      Subgroup:      Date:      **Lab instructor signature:**

Names:

## Results

Phase	E.P (mL)	N (I <sub>2</sub> ) (N)	K
H <sub>2</sub> O			
CHCl <sub>3</sub>			

***Homework:***

1. What indication does the partition coefficient value give regarding iodine solubility?
2. What type of reaction occurs between iodine and sodium thiosulfate during the titration?
3. Why doesn't this titration need an indicator?
4. Why is potassium iodide added to the organic layer before titration with sodium thiosulfate?

## LAB. 2

# COMPLEXATION

### Introduction

Complexes or coordination compounds results from a donor–acceptor mechanism or Lewis acid–base reaction between two or more different chemical constituents. Complexes can be divided broadly into two classes depending on whether the acceptor component is a *metal ion* or an *organic molecule*.

Intermolecular forces involved in the formation of complexes are: (1) Van der Waals forces, (2) Hydrogen bonding, (3) coordinate covalence (important in metal complexes), (4) charge transfer, and (5) hydrophobic interaction.

The stability of a complex in solution is expressed quantitatively by the *stability constant* (or *formation constant*) which is a measure of the strength of the interaction between the two species involved in the complex.

The stability constant for complexes can be determined by several methods, such as: (1) continuous variation method, (2) pH titration method, (3) solubility method, and (4) distribution method.

The aim of this experiment is to determine the stability constant of iodine -iodide complex by the distribution method.

### Materials and Equipment

1. Iodine 5% (in  $\text{CHCl}_3$ ), potassium iodide 0.1 N and 10%, and sodium thiosulfate 0.1 N.
2. Separatory funnel, burette, conical flasks, and graduated cylinder.

### Procedure

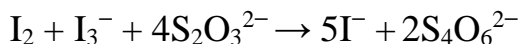
1. In dry stoppered separatory funnel, put 20 ml of 1% iodine (in  $\text{CHCl}_3$ ).
2. Add 20 ml of potassium iodide 0.1 N solution to the funnel.
3. Shake the flask for 15 min until equilibrium is established, allow to stand for 15 min until the phases is completely separated.
4. Separate the organic layer form the aqueous layer
5. Withdraw 10 ml of the aqueous layer and titrate against 0.1 N sodium thiosulfate until the light brownish color disappear.
6. Draw 5 ml of the organic layer, add 5 ml of 10% potassium iodide, and titrate against 0.1 N sodium thiosulfate until the light brownish color disappear.

## Calculations

Because of the presence of KI, iodine is solubilized in the form of soluble complex, therefore the chloroform layer contains only iodine while that of the aqueous layer contains iodine, iodide and iodine–iodide complex ( $I_3^-$ )

### Aqueous phase:

Both iodine ( $I_2$ ) and iodine–iodide complex ( $I_3^-$ ) will react with sodium thiosulfate in the aqueous layer:



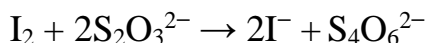
$$N_1 \times V_1 = N_2 \times V_2$$

$$I_2 + I_3^- \quad S_2O_3^{2-}$$

$$N(I_2 \text{ H}_2\text{O} + I_3^- \text{ H}_2\text{O}) \times 10 = 0.1 \times E.P$$

### Organic phase:

Only iodine ( $I_2$ ) will react with sodium thiosulfate in the organic layer:



$$N_1 \times V_1 = N_2 \times V_2$$

$$I_2 \quad S_2O_3^{2-}$$

$$N(I_2 \text{ CHCl}_3) \times 5 = 0.1 \times E.P$$

Partition coefficient (K) is constant for iodine and is determined in Lab.1. Therefore; the concentration of iodine in water is determined from partition coefficient and the concentration of iodine in chloroform layer:

$$K = N(I_2 \text{ H}_2\text{O}) / N(I_2 \text{ CHCl}_3)$$

$$N(I_2 \text{ H}_2\text{O}) = K \times N(I_2 \text{ CHCl}_3)$$

The concentration of  $I_3^-$  in water is obtained from:

$$N(I_3^- \text{ H}_2\text{O}) = N(I_2 \text{ H}_2\text{O} + I_3^- \text{ H}_2\text{O}) - N(I_2 \text{ H}_2\text{O})$$

The concentration of remaining  $I^-$  in water is obtained by subtracting the amount of  $I^-$  used in the formation of the complex (which is equal to  $N(I_3^- \text{ H}_2\text{O})$ ), from the initial concentration of  $I^-$  (which is equal to 0.1 N of KI):

$$N(I^- \text{ H}_2\text{O}) = 0.1 - N(I_3^- \text{ H}_2\text{O})$$

And the formation rate ( $K_f$ ) constant is obtained from:

$$K_f = \frac{N(I_3^- \text{ H}_2\text{O})}{N(I^- \text{ H}_2\text{O}) \times N(I_2 \text{ H}_2\text{O})}$$



Group:      Subgroup:      Date:      **Lab instructor signature:**

Names:

## Results

Phase	E.P. (mL)	N ( $I_2$ )	N ( $I_3$ )	N ( $I^-$ )	$K_f$
H <sub>2</sub> O					
CHCl <sub>3</sub>					



## ***Homework***

What are the pharmaceutical applications of complexation?



## LAB. 3

# CHEMICAL KINETICS

### Introduction

Kinetics is the study of rate reaction and factors affecting this rate.

Rate is the speed of a reaction. It is given by  $\pm (dc/dt)$ . This expression gives the increases (+) or decrease (–) of a concentration  $c$  within a given time interval  $dt$ .

Order of reaction is the number of atoms or molecules whose concentration determine the rate of reaction.

Types of chemical reaction:

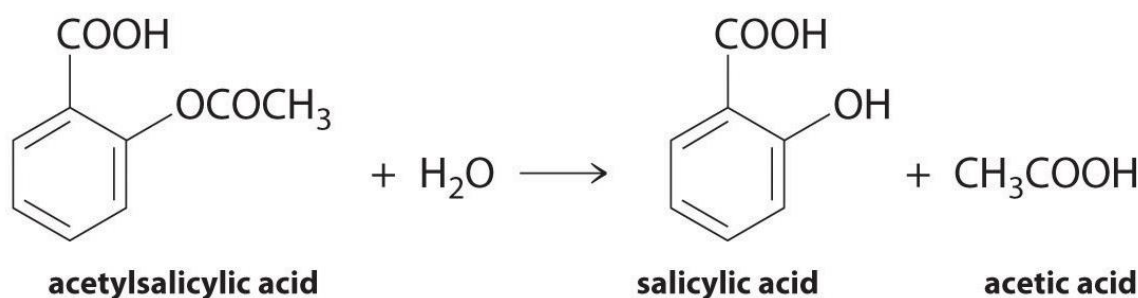
1. Zero order reaction
2. First order reaction
3. Second order reaction
4. Third order reaction

Half-life is the time required for the drug to decompose to one half the original concentration.

Shelf-life is the time required for the drug to lose 10% of its original concentration.

Experimental work

In this experiment we study the effect of temperature on the hydrolysis of aspirin which follows first order kinetics in which:



$$\ln C_t = \ln C_0 - Kt$$

First order reaction equation, it is a straight line equation

Thus, a plot of  $\ln C_t$  versus time will give a straight line.

$$\text{Slope} = -K$$

$K$  = Reaction rate constant ( $\text{min}^{-1}$ )

$$K = -\text{Slope}$$

The intercept of the line is equal to  $\ln C_0$

Half-life for 1<sup>st</sup> order reaction  $t_{0.5} = 0.693/K_{25^\circ\text{C}}$

Shelf-life for 1<sup>st</sup> order reaction  $t_{0.9} = 0.105/K_{25^\circ\text{C}}$

According to accelerated storage test the  $K$  values for the hydrolysis of aspirin at various elevated temperatures (40, 55, 70 °C) are obtained by plotting  $\ln C_t$  against time (t).

The logarithms of the specific rates of decomposition are then plotted against the reciprocals of the absolute temperatures and the resulting line is extrapolated to room temperature.  $K_{25^\circ\text{C}}$  is used to obtain a measure of the stability of the drug under ordinary shelf conditions. The plot is known as Arrhenius plot for predicting stability at room temperatures. Arrhenius found that the speed of many reactions increases about 2 or 3 times with each 10 °C rise in temperature. The effect of temperature on reaction rate is given by the equation known as Arrhenius equation.

Arrhenius equation, which expresses the relationship between the reaction rate constants and the temperatures at which they apply is used to find the rate of reaction at the temperatures of storage:

$$K = Ae^{-Ea/RT}$$

$$\ln K = \ln A - \frac{Ea}{R} * \frac{1}{T}$$

In which  $K$  is the specific reaction rate,  $A$  is a constant known as frequency factor,  $Ea$  is the energy activation,  $R$  is the gas constant (1.987 cal/deg.mol), and  $T$  is the absolute temperature.

## Materials and Equipment

- Aspirin, trisodium citrate, 0.05 N NaOH, Phenol red indicator
- Conical flasks, pipette, burette, water bath

## Procedure

1. Prepare the following mixture in a 250 mL conical flask: 4.5 g Aspirin, 9 g tri-sodium citrate, distilled water to 250 mL.



2. Take 10 mL from solution and titrate with 0.05 N NaOH solution using phenol red as indicator. The end point is a change from yellow to pink. This end point at 0 time is represented by x which is the volume of NaOH which is equivalent to aspirin before hydrolysis.
3. Label 3 flasks with the experimental temperature 40, 55, 70 °C and place about 80 mL of the mixture in each.
4. Note the time and place the flasks for the elevated temperature in the water baths provided.
5. Take 10 mL sample from each flask every 15 min for one hour. Titrate the samples with 0.05 N NaOH solution
6. Find the end point for each temperature at the time interval mentioned and represent them (y1, y2, y3, y4) mL of 0.05 NaOH.

### Calculation

1. Take the ln of C% and plot it against time (min) for each temperature.
2. Find the rate constant of each temperature from the slope of the line
3. Take lnK and plot it against time (1/T) (Arrhenius plot) to find K at 25 °C,  $t_{0.5}$  (half-life) and  $t_{0.9}$  (shelf-life).

Time	mL NaOH	C%	logC%

At 0 time Aspirin is not hydrolyzed yet:

E.P.<sub>0</sub> = ml of NaOH equivalent to Aspirin

$$[\text{NaOH}] * V_{\text{NaOH}} = [\text{Aspirin}] * V_{\text{Aspirin}}$$

$$0.05 * \text{E.P.}_0 = [\text{Aspirin}] * 10$$

After  $t$  min Aspirin is hydrolyzed forming salicylic acid and acetic acid

E.P.<sub>t</sub> = ml of NaOH equivalent to (remaining Aspirin + salicylic acid + acetic acid)

**Aspirin**  $\longrightarrow$  **Acetic acid + Salicylic acid**

$$[\text{Aspirin}] \quad \quad \quad 0 \quad \quad \quad 0$$

$$[\text{Aspirin}] - X_t \quad \quad \quad X_t \quad \quad \quad X_t$$

$$0.05 * \text{E.P.}_t = ([\text{Aspirin}] - X_t + X_t + X_t) * 10$$

$$0.05 * \text{E.P.}_t = ([\text{Aspirin}] + X_t) * 10 \dots \dots \dots (1)$$



Where  $X_t$  represents the concentration of degraded aspirin at time  $t$

$$\% \text{ [remaining Aspirin]} = \frac{[\text{Aspirin}] - X_t}{[\text{Aspirin}]} * 100 \dots\dots\dots (2)$$

Use equations (1) and (2) to find (% [remaining Aspirin]) in the 10 ml samples for the remaining time intervals

Draw a linear graph of  $\ln \% \text{ [remaining Aspirin]}$  against time (min) for each temperature and determine the reaction rate constant  $K$  from the slope:

$$\text{Slope} = -K$$

Draw a linear graph of  $\ln K$  against the reciprocal of temperature  $1/T$  ( $\text{min}^{-1}$ ) (Arrhenius plot) to find  $K$  at  $25^\circ\text{C}$ ,  $t_{0.5}$  (half-life) and  $t_{0.9}$  (shelf-life).

Time (t) (min)	E.P. (ml)	$X_t$ (N)	$[\text{Aspirin}] - X_t$ (N)	% [remaining Aspirin]	$\ln \% \text{ [remaining Aspirin]}$
0					
15					
30					
45					
60					

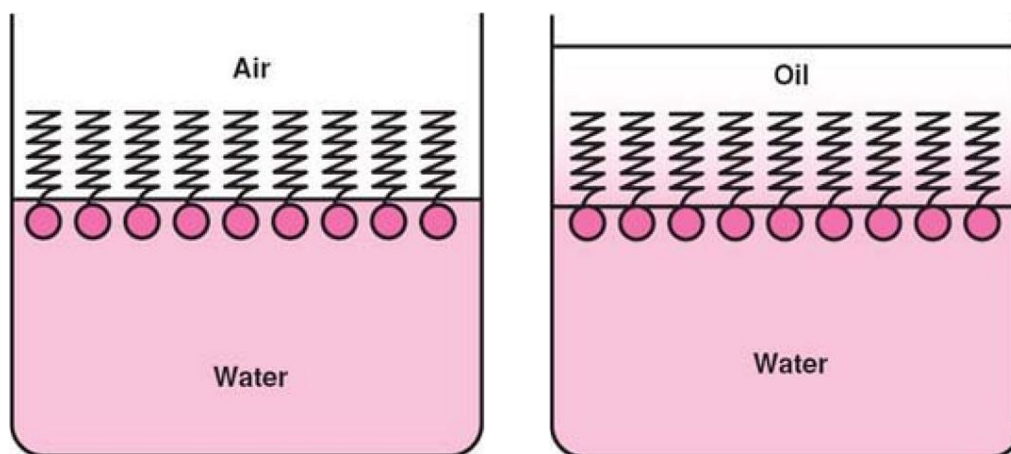
Temp.(T) ( $^\circ\text{C}$ )	$t$ (min)	$1/t$ ( $\text{min}^{-1}$ )	$K$	$\ln K$	$t_{0.5}$ (min)	$t_{0.9}$ (min)
25						
40						
55						
70						

## LAB. 4

# SURFACTANTS

### Introduction

Molecules and ions that are adsorbed at interfaces are termed *surface-active agents* or *surfactants*. An alternative term is *amphiphile*, which suggests that the molecule or ion has a certain affinity for both polar and nonpolar solvents. When such molecule is placed in an air-water or oil-water system, the polar groups are attached or oriented toward the water, and the nonpolar groups are oriented toward the air or oil (Figure 1).



**Figure 1:** Adsorption of fatty acid molecules at a water–air interface (left panel) and a water–oil interface (right panel).

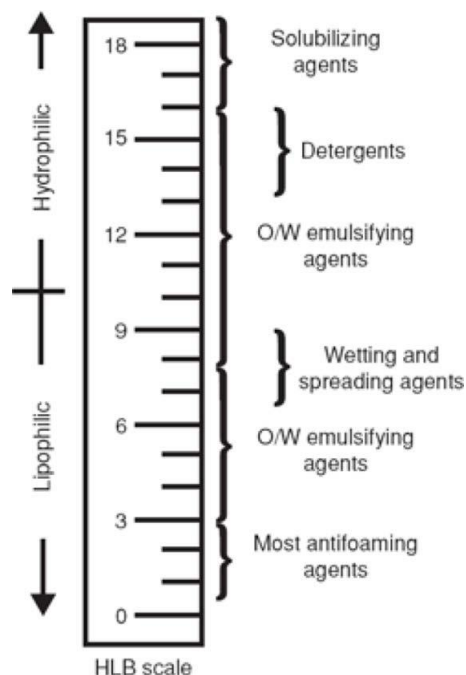
Surface active agents are used in many pharmaceutical preparations as wetting agents, emulsifiers, solubilizers and antifoaming agents.

### HLB system

The hydrophile-lipophile balance or HLB system is an arbitrary scale that has been set up for expressing the hydrophilic and lipophilic characteristics of an emulsifying agent. Agents with HLB value of 1-8 are lipophilic and suitable for preparation of w/o emulsion, and those with HLB value of 8-18 are hydrophilic and good for o/w emulsion (Figure 2). The HLB values of some commonly used emulsifying agents are given in Table 1.

The oil phase of an oil-in-water (O/W) emulsion requires a specific HLB, called the *required hydrophile–lipophile balance* (RHLB). A different RHLB is required to form a water-in-oil (W/O) emulsion from the same oil phase. The

RHLB values for both O/W and W/O emulsions have been determined empirically for a number of oils and oil-like substances, some of which are listed in Table 2.



**Figure 2:** A scale showing surfactant function on the basis of hydrophilic–lipophilic balance (HLB) values.

**Table 1:** HLB values for selected emulsifiers

AGENT	HLB
Ethylene glycol distearate	1.5
Sorbitan tristearate (Span 65)	2.1
Propylene glycol monostearate	3.4
Triton X-15	3.6
Sorbitan monooleate (Span 80)	4.3
Sorbitan monostearate (Span 60)	4.7
F Diethylene glycol monolaurate	6.1
Sorbitan monopalmitate (Span 40)	6.7
Sucrose dioleate	7.1
Acacia	8.0
Amercol L-101	8.0
Polyoxyethylene lauryl ether (Brij 30)	9.7
Gelatin	9.8
Triton X-45	10.4
Methylcellulose	10.5
Polyoxyethylene monostearate (Myrj 45)	11.1
Triethanolamine oleate	12.0
Tragacanth	13.2
Triton X-100	13.5
Polyoxyethylene sorbitan monostearate (Tween 60)	14.9
Polyoxyethylene sorbitan monooleate (Tween 80)	15.0



Polyoxyethylene sorbitan monolaurate (Tween 20)	16.7
Pluronic F 68	17.0
Sodium oleate	18.0
Potassium oleate	20.0
Sodium lauryl sulfate	40.0

**Table 2:** Required HLB for some Oil-Phase ingredients for oil-in water (O/W) and water-in-oil (W/O) emulsions

SUBSTANCE	O/W	W/O	SUBSTANCE	O/W	W/O
Cottonseed oil	6–7	—	Kerosene	12–14	—
Petrolatum	8	—	Cetyl alcohol	13–16	—
Beeswax	9–11	5	Petroleum ether	14	—
Paraffin wax	10	4	Stearyl alcohol	15–16	—
Mineral oil	10–12	5–6	Carbon tetrachloride	16	—
Methyl silicone	11	—	Lauric acid	16	—
Lanolin, anhydrous	12–14	8	Oleic acid	17	—
Carnauba wax	12–14	—			
Lauryl alcohol	14	—			
Castor oil	14	—			

### *Calculation of HLB value for oil-in-water emulsions*

1. Determine the RHLB for the oil phase of an oil-in-water (O/W) emulsion. For example consider 100 ml (O/W) emulsion consists of a mineral oil with a RHLB of 10.6.
2. Chooses a blend of two emulsifying agents, one with an HLB above and the other with an HLB below the required HLB of the emulsion (RHLB = 10.6 in this example). From Table 1, Tween 80 (Polyoxyethylene 20 sorbitan monooleate), with an HLB of 15, and Span 80 (sorbitan monooleate), with an HLB of 4.3 was chosen.
3. Calculate the percentages of the emulsifying agents. The formula for calculating the percentage of Tween 80 (surfactant with the higher HLB) is

$$\% \text{ Tween} = \frac{\text{RHLB} - \text{HLB low}}{\text{HLB high} - \text{HLB low}}$$

Where HLB high is for the higher value, 15, and HLB low is for the lower value, 4.3.

$$\% \text{ Tween} = \frac{10.6 - 4.3}{15 - 4.3} = 0.59$$



5 ml of emulsifier has been estimated as proper protection for the O/W emulsion. Therefore,  $5 \text{ ml} \times 0.59 = 2.95 \text{ ml}$  of Tween 80 is needed and the remainder, 2.05 ml, must be supplied by Span 80 for the 100 ml emulsion.

### **Materials and equipment**

- Stearic acid, castor oil, span 80, tween 80, and water.
- Beaker, dropper, graduated cylinder, volumetric flask, and spatula.

### **Procedure**

#### **Demonstration of the surface orientation of surfactant.**

1. Place approximately 5 g of stearic acid on the surface of hot water in a beaker, the fatty acid will melt to a lens-shape drop.
2. Allow the water to cool. When the stearic acid has solidified, remove it without disturbing the surface and allow it to dry.
3. Put few drops of water on the top and the bottom of the cake. Record your observation.

#### **Preparation of 100 ml of 10% castor oil o/w emulsion by using a 3% blend of Span 80 and Tween 80.**

1. From Table 2 determine the RHLB for castor oil o/w emulsion.
2. Calculate the proportion and the amount of Span 80 and Tween 80 necessary to provide the required HLB. Use Table 1 to find the HLP values for these emulsifiers.
3. Transfer the amount of water into a 100 ml volumetric flask, and add the water soluble emulsifier (Tween 80) to the water. Shake the flask well.
4. Add the oil soluble emulsifier (Span 80) to 10 ml castor oil in a 100 ml beaker and stir thoroughly.
5. Add the oil phase in portions to the aqueous phase in the flask and thoroughly shake the mixture in the capped container.

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## ***Results***

### **Demonstration of the surface orientation of surfactant**

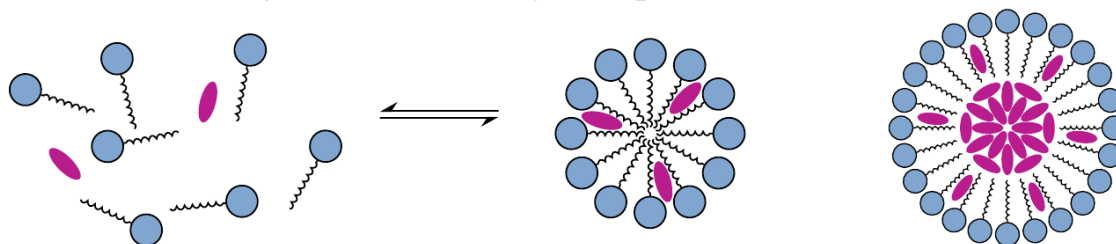
### **Preparation of castor oil o/w emulsion**

## LAB. 5

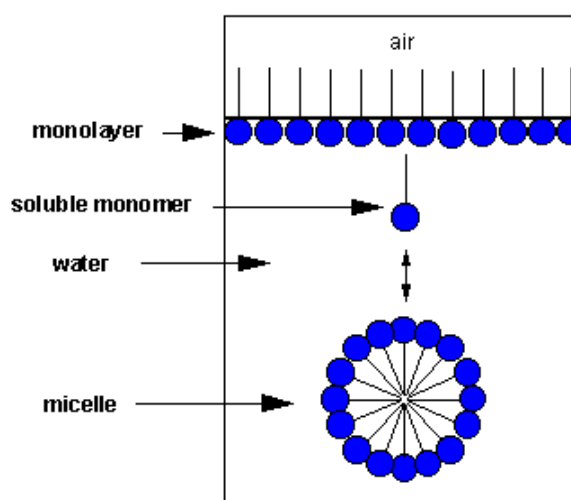
# SOLUBILIZATION BY SURFACTANTS

### Introduction

Solubilization by surfactants is the process where water-insoluble substances are brought into solution by incorporation into micelle.



When surfactants present in a liquid medium at low concentration, they exist separately (mostly in the interface or surface of liquid). When the concentration of a surfactant reaches a given concentration where the surface is saturated with surfactant molecules, aggregation in the bulk of the liquid occurs. These aggregates are called “micelles”; and the concentration at which they are formed is called critical micelle concentration (CMC).



Most micelles are spherical and contain between 60 and 100 surfactant molecules. In a micelle, polar or ionic heads form an outer shell in contact with water, while non polar tails are sequestered in the interior to avoid water and obtain minimum energy state. The typical micelle diameter is about 2–3 nm; therefore, they are not visible under the light microscope.



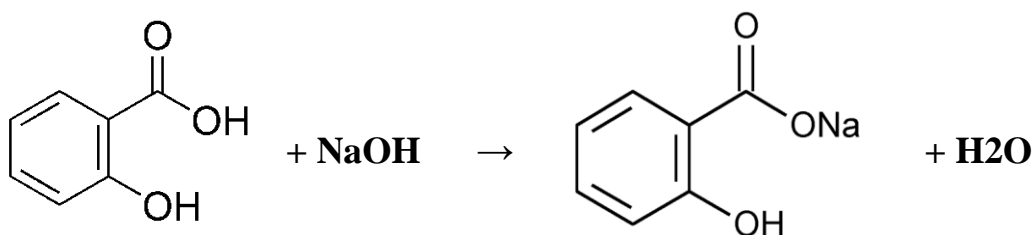
Micelles can increase the solubility of slightly soluble drugs (hydrophobic drugs) by incorporating them into the hydrophobic core of the micelle. Solubilization does not occur until the micelles are formed (i.e. surfactant concentration must be above CMC). The amount of substance solubilized increases as the number of micelles increases.

The aim of this experiment is to observe the effect of increasing the concentration of Tween 80 (a hydrophilic surfactant) on the solubility of salicylic acid (a slightly soluble weak acid).

### Procedure

1. Prepare 6 solutions of 0.25 g of salicylic acid in 25 ml of different concentrations of Tween 80 (0%, 1%, 2%, 4%, 6%, and 10%).
2. Shake the flasks for 10 minutes, then filter.
3. Titrate 10 ml of the filtrate against NaOH solution (0.05 N) using phenol red as indicator. The end point is determined when the color changes from yellow to pink.
4. Plot the solubility (w/v %) of salicylic acid against concentration (w/v %) of tween 80.

### Calculations



$$N_{\text{NaOH}} \times V = \frac{\text{wt Salicylic acid}}{\text{Eq. wt Salicylic acid}}$$

$$0.05 \times 0.001 = \frac{\text{wt Salicylic acid}}{138.1}$$

$$\text{Wt Salicylic acid} = 0.0069 \text{ g salicylic acid}$$

0.0069 is the chemical factor for salicylic acid in this titration; which is the number of grams of substance that is equivalent to 1 ml of the standard solution.

$$\text{Chemical factor} \times \text{end point} = \text{g salicylic acid in 10 ml}$$

### Material and equipment

- Salicylic acid, Tween 80, DW, phenol red indicator, and NaOH
- Volumetric flask (25 ml), conical flask (50 ml), pipettes, burette, filter paper, and funnel.

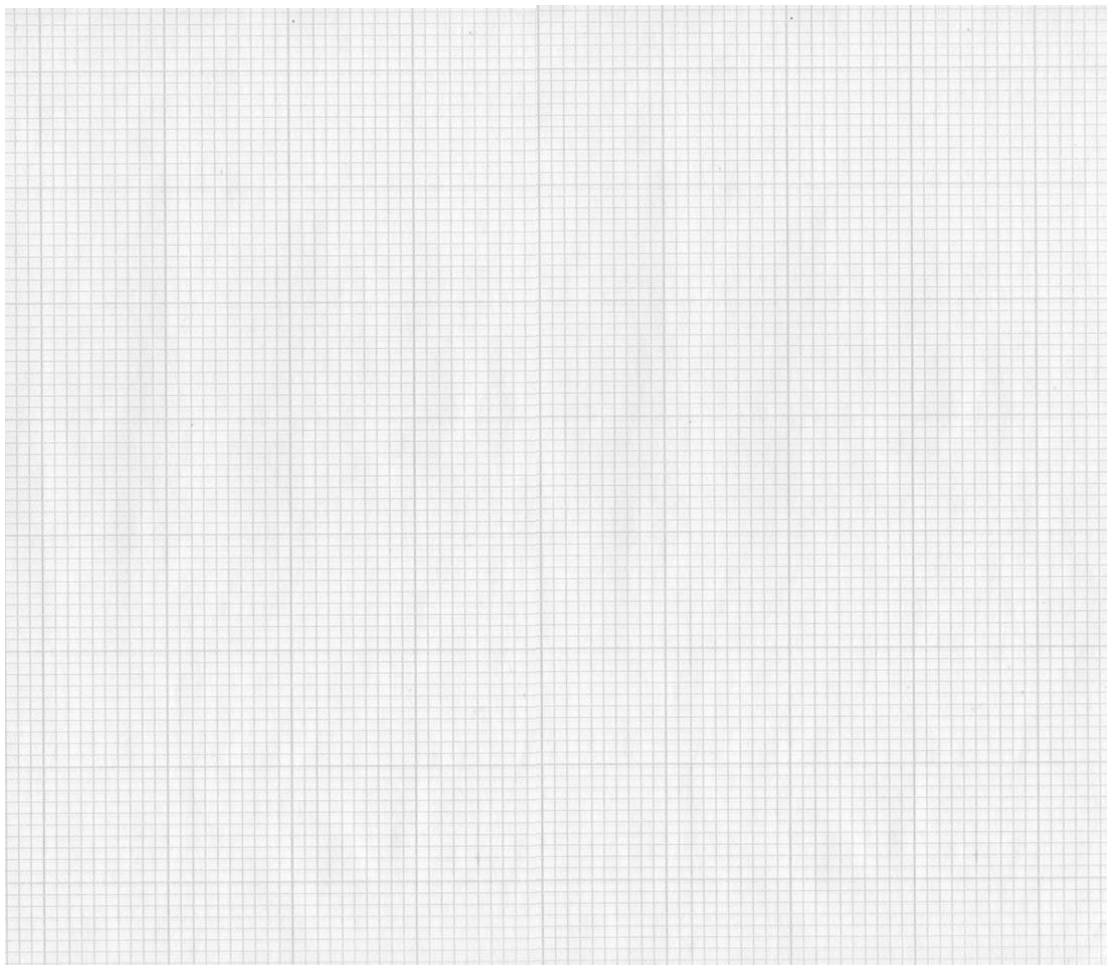
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Names:

## Results

Flask	Tween 80 conc. (w/v %)	E.P. (ml)	Salicylic acid chemical factor (g/ml)	Salicylic acid Solubility (g/10 ml)	Salicylic acid Solubility (w/v %)
1	0				
2	1				
3	2				
4	4				
5	6				
6	10				

## Graph



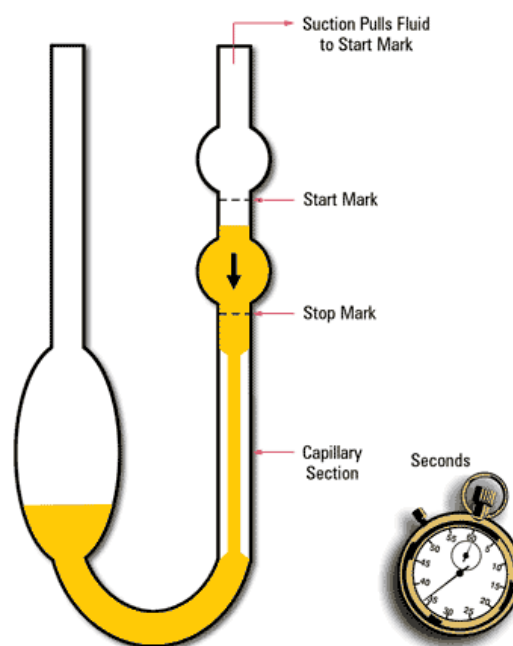
## LAB. 6

# VISCOSITY

### Introduction

Viscosity is an expression of the resistance of a fluid to flow; the higher the viscosity, the greater is the resistance. The unit of viscosity is the poise (p) and centipoise (cp).

Capillary viscometer is used for the determination of the viscosity of Newtonian systems (systems with single viscosity values). It is a u-shaped glass apparatus with a wide arm and a narrow one that contain a capillary tube. There are two marks in the narrow part; above and below the bulb. By this method the viscosity of an unknown liquid ( $\eta_1$ ) can be determined in relation to another liquid of known viscosity ( $\eta_2$ ).



Einstein developed an equation that relates the *intrinsic* viscosity ( $\eta$ ) of dilute colloidal dispersions of spherical particles with the volume fraction of colloidal particles present ( $\phi$ ):

$$\eta = \eta_0(1 + 2.5\phi)$$

$\eta_0$ : viscosity of the dispersion medium.

Several viscosity coefficients can be defined with respect to this equation. These include *intrinsic viscosity* ( $\eta$ ), *relative viscosity* ( $\eta_{rel}$ ), and *specific viscosity* ( $\eta_{sp}$ ):

$$\eta_{rel} = \frac{\eta}{\eta_0} = 1 + 2.5\phi$$

$$\eta_{sp} = \frac{\eta}{\eta_0} - 1 = 2.5\phi$$

In this experiment, the concentration (volume fraction  $\phi$ ) of an unknown liquid is determined by measuring its relative viscosity ( $\eta_{rel}$ ).

### Materials and equipment:

- 50% w/v glycerin solution, water.
- Capillary viscometer, stop watch, volumetric flask (50 mL), graduated cylinder, sucker, and balance.



## Procedure

1. Prepare 50 mL solutions of different volume fractions ( $\phi$ ) of glycerin in water: 0.05, 0.1, 0.15, 0.2, and 0.25 from 0.5 glycerin solution (stock solution).
2. Measure the viscosity of the solutions by capillary viscometer:
  - a. Fill the viscometer with water until its level reaches the stop mark.
  - b. Using a sucker bulb, suck the water from the stop mark to the start mark. After that, put your finger on the tip of the viscometer to prevent the water from going down.
  - c. Leave the water to descend from the start mark to the stop mark and record the time.
3. Repeat the above procedure for the unknown liquid.

## Calculation

1. The viscosity (in cp) of the liquid under test ( $\eta_1$ ) can be found by measuring the time (in sec) ( $t_1$ ) required for this liquid to pass between the two marks as it flows by gravity through the vertical capillary tube. This time is compared with the time ( $t_2$ ) required for a liquid of known viscosity ( $\eta_2$ ) (usually water) to pass between the two marks.

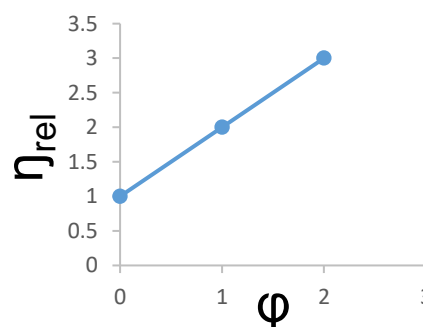
$$\eta_{rel} = \frac{\eta_1}{\eta_2} = \frac{\rho_1.t_1}{\rho_2.t_2}$$

$P_1$  and  $P_2$  are the densities of the measured liquid and water respectively.

The densities of the prepared solutions are: 1.005, 1.018, 1.03, 1.037, 1.044 respectively.

2. The measured relative viscosities for the prepared solutions ( $\eta_{rel}$ ) are plotted against their volume fraction ( $\phi$ ) to obtain a straight line with an intercept of 1 (viscosity of water), according to Einstein equation:

$$\eta_{rel} = 1 + 2.5\phi$$



3. The volume fraction  $\phi$  of the unknown liquid can be determined from the curve by calculating its relative viscosity  $\eta_{rel}$
4. The density of the unknown liquid can be determined by dividing the mass of 50 mL of the liquid by its volume (use graduated cylinder).



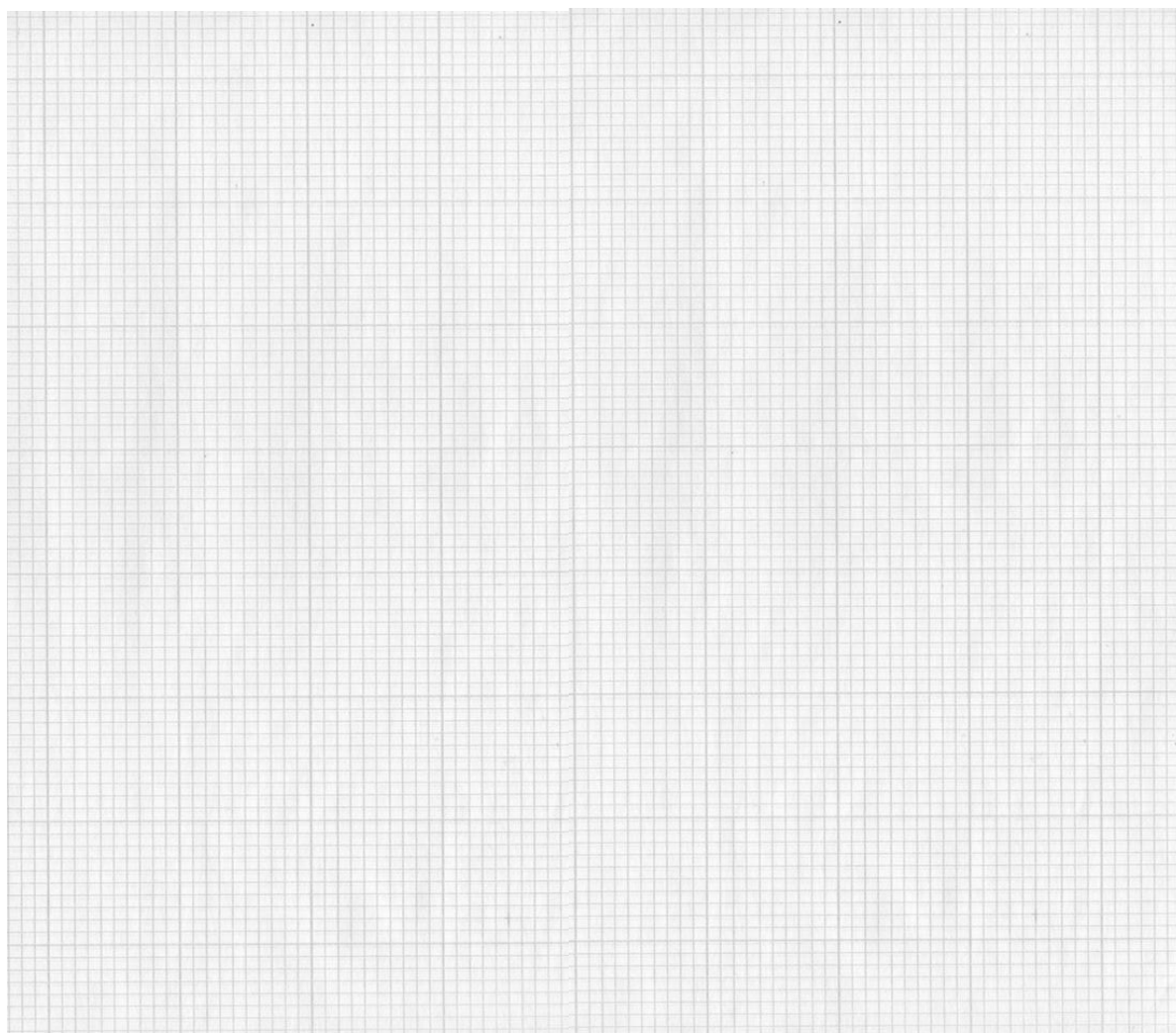
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## Results

Flask No.	Glycerin ( $\phi$ )	$P_2$	$t_2$ (sec)	$\eta_1$ (cp)
1	0.05	1.005		
2	0.1	1.018		
3	0.15	1.030		
4	0.2	1.037		
5	0.25	1.044		
Unknown				

## Graph



# REFERENCES

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