



S.A.RAJA PHARMACY COLLEGE

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SUBJECT:
PHYSICAL PHARMACEUTICS

III SEMESTER B.PHARM

PRACTICAL LAB MANUAL

1. DETERMINATION OF SOLUBILITY OF DRUG AT ROOM TEMPERATURE

Aim:

To determine the solubility of inorganic salt at room temperature

Apparatus required:

- Pure inorganic salt
- Distilled water
- Porcelain dishes (or) watch glasses.
- Beaker
- Pipette(10 ml)

Principle:

Solubility is defined in quantitative terms as the concentration of solute in a saturated solution at a certain temperature qualitatively it is defined as a spontaneous interaction of two (or) more substances to form a homogeneous molecular dispersion.

Quantitatively solubility is expressed in molarity, Molarity and percentage (w/v (or) w/w). a saturated solution is defined as the one in which the solute is in equilibrium with the solid phase (solute) at a certain temperature.

A solution may sometimes contain more amount of solute than in saturated one, such a solution is known as super saturated solution

The solubility of the solid depends on temperature, melting point of the solid and molar heat of fusion.

Procedure:

1. Clean all glasswares using detergent solution and chromic acid solution.
2. Wash two (or) three times using purified water.
3. Take 50ml of distilled water in a beaker (100).
4. Add some amount of NaCl in distilled water and stir using glass rod (or) by an electric motor driven shaker.
5. Take sample using a pipette with a piece of filter paper, tie at the tip of the pipette. So that the insoluble salt can be removed.
6. Remove the piece of the filter paper from the tip of the pipette and transfer 10l of the solution in weighed porcelain dish (or) watch glass used in direct heat (or) on the water bath (or) micro oven.
7. Dry the solution till constant weight.
8. Take weight of the dish using double pan balance (or) chemical balance calculate weight of the powder.

9. Record all the parameter in the form of table are in proper way of that it becomes easy to understand the concept of solubility.

Calculation:

Temperature	Weight of empty dish (w ₁) gm	Weight of empty dish with solution(w ₂) gm	Weight of dish with residue (w ₃) gm	Weight residues (w ₃ - w ₁) gm	Weight of solvent (w ₂ - w ₃) gm	Solubility in g/100

$$\text{Solubility of salt} = \frac{\text{Weight of residue}}{\text{Weight of solvent}} * 100$$

$$\frac{W3 - W1}{W2 - W3} * 100$$

Report:

Solubility of the given salt at a room temperature was found to be –

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

(i) Drop count method

Aim:

To determine the surface tension of a give liquid using stalagmometer by drop count method.

Requirements:

- Stalagmometer
- Glycerin
- Water
- Specific gravity bottle
- Balance

Procedure:

- Select a clean and dry stalagmometer than fix firmly in a vertical portion, in a beaker containing water and suck the water to the limb up to the level higher than the upper limb by using finger.
- Leave the finger and allow the under water to flow down start counting of drops when the meniscus passes through the upper mark and until passes the lower mark (the number of drops should be in between 15-20) repeat the experiment for 3 times.
- Remove water and clean the stalagmometer.
- Select the liquid and repeat above step with liquid.
- Density of water and liquid are measure., them substitute these values together with number of drops of water and liquid in the equation and calculate the surface tension.

Calculation:

Weight of empty bottle (W_1) =

Weight of bottle with water (W_2) =

Weight of water (WW) = $W_2 - W_1$

LIQUID :

Weight of the empty bottle with liquid (W_3) =

Weight of liquid = $W_3 - W_1$ =

$$\text{Density of water } \left(\frac{g}{v}\right) = \frac{W1 - W2}{V}$$

S.no	Name of liquid	Number of drops			Mean
1.	Water				
2.	liquid				

Surface tension of water $\gamma_1 =$

Number of drops of water $n_1 =$

Number of drops of liquid $n_2 =$

$$\left(\frac{\gamma_2}{\gamma_1}\right) = \frac{n_1 \rho_2}{n_2 \rho_1}$$

$$\gamma_2 = \frac{n_1 \rho_2 \gamma_1}{n_2 \rho_1}$$

Report:

Surface tension of the given liquid $\gamma_2 =$

3. DETERMINATION OF SURFACE TENSION OF LIQUID BY DROP WEIGHT METHOD

Aim:

To determine the surface tension of given liquid by drop weight method.

Requirement:

- Stalagmometer
- Glycerin
- Water
- Balance
- Weighing bottle

Procedure:

- The clean stalagmometer is fixed with a clamp and filled with water by sucking.
- Around 20 to 30 drops of water falling from the stalagmometer. This is collected in a dry weighing bottle and weighted.
- The stalagmometer is then dried and experiment is repeated with the liquid. Whose surface tension to be determined.
- Care should be taken during experiments that the number of drops does not exceed 20 per minutes.

Calculation:

Weight of china dish (X_1) =

Weight of china dish + water (Y_1) =

Weight of china dish + liquid (Y_2) =

Weight of 20 drops of water = $Y_1 - X_1$

Weight of one drop of water (W_1) = $\frac{Y_1 - X_1}{20}$

Weight of 20 drop of liquid = $Y_2 - X_1$

Weight of one drop of liquid (W_2) = $\frac{Y_2 - X_1}{20}$

Surface tension γ = ?

$$\frac{W_1}{W_2} * \gamma_2$$

γ_2 = --- mN/m

Report:

Surface tension of a given liquid $\gamma = \text{--- mN/m}$.

4. DETERMINATION OF PARTITION CO-EFFICIENT OF IODINE IN CCL₄ AND WATER

Aim:

To establish the partition co-efficient of iodine in distilled water and carbon tetra chloride.

Requirements:

Chemicals:

- Distilled water
- Iodine crystals
- Carbon tetra chloride
- 0.01N Na₂S₂O₃
- Starch indicator
- 0.1N Na₂S₂O₃

Apparatus:

- Separating funnel
- Conical flask
- Pipette
- Burette
- Reagent bottles

Principle:

When a solute is shaken with two immiscible solvents it gets partitioned between the solvents. This distribution of solute in two solvents depends upon the solubility of the solute in two solvents.

At the distribution equilibrium, the ratio of concentrations of solute in both solvents is constant at a given temperature.

The constant is called the partition co-efficient (Or) distribution co-efficient of the solute between the two solvents.

Note:

Carbon tetra chloride is 1.6 times heavier than water when mixed the two liquids the water floats on the surface of the tetra chloride forming clear layers and can be easily separated.

Procedure:

- Approximately prepare 110ml of saturated solution of iodine in CCl₄ for use as stock solution.
- Prepare the following mixtures in separating funnels.

Set I : 50ml water + 40ml stock solution + 10ml ccl₄

Set II: 50ml water + 30ml stock solution + 20ml ccl₄

Set III: 50ml water + 20ml stock solution + 30ml ccl₄

Set IV: 50ml water + 10ml stock solution + 40ml ccl₄

- Shake the mixture in separating funnel force fullyfor about30 minutes for the iodine to get distributed between the two solventsandto reach the distribution wquilibrium.
- Let the flask to stand for about 10 minutes. So thattow clear layers are separated.
- Now remove stopper of a separating funnel and keep its mouth open during this period to facilitate the separation.
- Release the lower layer which is occurs into four different stoppered dry bottles (discard the immediate layer between both the phases).
- Organic layer stays in the separating funnel. Using a dry pipette withdraw 10ml of organic layer to a conical flask.
- Titrate it against 0.01N Na₂S₂O₃, starch is used as indicator.
- End point is disappearance of blue colour.
- Withdraw 10 ml of aqueous layer using a dry pipette and titrate it against 0.01N Na₂S₂O₃, using starch as an indicator.
- Repeat the process for all the mixture

S.no	Content of the flask	Burette reading		Volume of sodium thiosulphate	Indicator	End point
		Initial	final			
1.	10ml aqueous layer				Starch indicator	Disappearance of blue colour
2.	10ml organic layer					

Calculation:

a) FOR AQUEOUS LAYER:

Concentration of iodine in aqueous layer

$$N_1 V_1 = N_2 V_2$$

$$N_2 = \frac{N_1 V_1}{V_2} * \text{equivalent weight factor}$$

Weight of sodium thiosulphate $N_1 = 0.01 \text{ N}$

Volume of sodium thiosulphate $V_1 =$ ml

Volume of aqueous layer layer $V_2 =$ ml

Normality of iodine I aqueous layer = ?

$N_2 =$

Report:

The partition co-efficient of ccl_4 and distilled water was found to be ----- moles.

5. DETERMINATION OF CRITICAL MICELLAR CONCENTRATION(CMC)

Aim:

To determine the critical micellar concentration of a surfactant (sodium lauryl sulphate) by surface tension measurement.

Principle:

The surface active agent (surfactant) concentrated at the surface of a (or) at the interface between two immiscible liquids (or) between a liquid under a solid they reduce the surface tension (or) interfacial tension as a total concentration of surface active agents in aqueous phase gradually increase the concentration of surface active agent under going absorption at the surface is increased until this result in gradually reduction of surface tension (or) interfacial tension.

At the one point both the surface and bulk of aqueous phase become saturated with monomer of surface active agent. This concentration point is CMC, at this point the surface (or) interfacial tension is reduced to the lowest value further the concentration of surface active agent will result in aggregation of monomers forming resulting without any appreciable change in surface tension. The surface tension Vs concentration curve for an aqueous solution of surfactant shows that progressive decrease in surface tension until the CMC is reached. The CMC is taken at the point of intersection of extra polluted straight line on either side of the break in the curve.

Apparatus and material required:

- Psychrometer
- Stalagmometer
- Sodium lauryl sulphate

Procedure:

- A stock solution of 5% SLS is prepared by dissolving 5gm of SLS (Sodium lauryl sulphate) in 100ml of water in a volumetric flask.
- A series of concentration is prepared by diluting stock solution of 0.5%, 1%, 1.5%, 2%, 2.5%, 3%, 4%, 4.5%, 5%.... surface tension of prepared solution is determined by drop count method.
- A graph of surface tension in X – axis and concentration in y – axis is plotted, the surface tension can be calculated for each concentration by using drop count method.

Surface tension

$$\gamma_2 = \frac{n_1 \rho_2}{n_2 \rho_1} * \gamma_1$$

Where,

Surface tension of water (γ_2) = dynes/cm

$n_1 \rightarrow$ Number of drops in water

$n_2 \rightarrow$ Number of drops in liquid

$\rho_1 \rightarrow$ Density of the water

$\rho_2 \rightarrow$ Density of the liquid

S.no	Sample	Concentration	Number of drops			Density	Surface tension
			I	II	average		
	water	0%					
	I	0.5%					
	II	1%					
	III	1.5%					
	IV	2%					
	V	2.5%					

Calculation:

WATER:

Weight of empty bottle (W_1) = gm

Weight of empty bottle with water (W_2) = gm

Weight of water (WW) = $W_1 - W_2$

Density of water (g/v) = $W_1 - W_2/V$

$$\rho_1 = \text{ gm/cm}^3$$

LIQUID:

Weight of empty bottle (W_1) = gm

Weight of empty bottle with liquid (W_2) = gm

Weight of liquid (WL) = $W_1 - W_2$

Density of liquid (g/v) = $W_1 - W_2/V$

$$\rho_2 = \text{ gm/cm}^3$$

Surfacetension

$$\gamma_2 = \frac{n_1 \rho_2}{n_2 \rho_1} * \gamma_1$$

$$\gamma_2 = \text{dynes /cm}$$

Report :

The critical micellar formation of sodium lauryl sulphate is determined.

6. DETERMINATION OF PKA VALUE BY HENDRSON HASSEL BALCH EQUATION

Aim:

To determine the dissociation constant Pka of a weak acid (salicylic acid) by hendrson hassel balch equation.

Principle:

Weak acid and weak base do not ionizes completely in aqueous solution like strong acid and bases. The degree of ionisation and dissociation constant (Pka) which express covalantly in the term of Pka. It is the negative logarithm of acid dissociation constant (Pka) is the same way as pH is used to represently negative logarithm of hydronium ion concentration.

It's dissociation constant value (Pka) for weak acid (or) base can be determined by using hendrson hassal balch equation.

$$\text{pH} = \text{Pka} + \log X \text{ (Ionised acid)/(unionized acid)}$$

$$\text{pH} = \text{Pka} + \log X \text{ (salt)/(acid)}$$

At 50% neutralization point that is when (ionized acid)=(unionized acid) the value of Pka-pH.

The value of pka can be derived by recording the pH at 50% neutralized point.

Materials required:

- Salicylic acid
- pH meter
- Burette
- Standard NaOH
- Conical flask
- Indicator

Procedure:

- 10ml of 0.5% w/v of salicylic acid in methanol is pipette out to be a conical flask.
- This is then titrated against 0.5% N NaOH solution using of methyl red is an indicator to complete neutralization.
- Burette reading is noted similarly 10ml of 0.5% W/V of salicylic acid is taked into a conical flask and titrated against standard 0.5N NaOH to 50% neutralisatiion point before the Pka value of salicylic acid at 30 ° C.

Calculation:

Let the volume of 0.5 N NaOH consumed in titration with 10ml of 0.5% salicylic acid solution.

At complete neutralization = X ml.

Volume of 0.5N NaOH required for half neutralization = X/2ml

pH of the half neutralization solution =

$$\text{pH} = \text{pK}_a + \log \frac{\text{X salt}}{\text{acid}}$$

Report:

The pK_a value of salicylic acid at 30°C is –

7. DETERMINATION OF VISCOSITY THE GIVEN UNKNOWN LIQUID

Aim:

To determine the viscosity of given unknown liquid.

Chemicals required:

- Oswald viscometer
- Relative density bottle
- Stand
- Unknown liquid
- Beaker
- Distilled water

Principle:

- The force of friction with the one part of the liquid offer to another part of liquid is called viscosity. For measuring the viscosity co-efficient oswald viscometer is used which is based on poise tuller's law.
- According to this law the rate of flow of liquid through a capillary tube having viscosity co-efficient (n) can be expressed as

$$N = \frac{\pi r^2 t p}{8 v l}$$

r → Radius of the capillary tube (cm).

n → Viscosity co-efficient (poise).

V → volume of liquid.

t → flow time in seconds through capillary

l → Length of capillary

p → Hydrostatic pressure.

- Since the hydro pressure.(driving force)of the liquid is given by,
- $P = dgh$

$$P = dgh$$

h → Height of the column.

d → Density of the liquid.

$N \propto Pt$

- If n_1 and n_2 are the viscosity coefficients of liquid D_1 and D_2 are the density of liquid as well as t_1 and t_2 are the time of flow of equal volume of liquid through the capillary respectively then

$$n_1 \propto d_1 g h t_1$$

$$n_2 \propto d_2 g h t_2$$

$$n_1/n_2 = d_1 g h t_1 / d_2 g h t_2$$

$$n_1/n_2 = d_1 t_1 / d_2 t_2$$

- Here, usually the viscosity of the given liquid is measured with respect to water whose viscosity is known as very accurately at different temperatures.
 - The SI unit of viscosity is pascal second.
- $\text{Pas} = \text{Kg m}^{-1} \text{s}^{-1}$
- The cgs unit for the same is poise. It is most commonly expressed as centipoises (cp = 0.001 pascal sec).
- Water at 20°C has a viscosity of 1.0020 cp.

Procedure:

- Wash the relative density bottle with distilled water and dry it. Take the weight of empty and filled relative density bottle with the given liquid clean and rinse the viscometer properly with distilled water.
- Fix the viscometer vertically in the stand and fill the specific amount of given unknown liquid in the viscometer.
- Time of flow is recorded when the liquid starts to flow from the mark C and D above and below the bulb A. The experiment is repeated 3-4 times.

Calculation:

S.no	Name of the liquid	Flow of liquid in second			Mean
		Trial 1	Trial 2	Trial 3	

Weight of an empty density bottle (w_1) =

Weight of the density bottle with water (w_2) =

Weight of the density bottle with unknown liquid (w_3) =

Weight of the water (w_w) = $w_2 - w_1$

Weight of the liquid (w_L) = $w_3 - w_1$

Relative density = Density of liquid/Density of water

Density of liquid (d_2) = mass/volume

Density of the water (d_1) = mass/volume

Viscosity of liquid $n_2 = e_2 t_2 / e_1 t_1 \times n_1$

Report:

The viscosity of the unknown liquid was found to be ----- centipoises.

8. DETERMINATION OF PERCENTAGE ADSORPTION OF OXALIC ACID BY CHARCOAL METHOD

Aim:

To determine the percentage adsorption of oxalic acid by charcoal method.

Requirement:

- Activated charcoal
- N/10 oxalic acid solution
- N/10 NaOH
- Phenolphthalein
- Pipette
- Burette
- Conical flask

Principle:

- The phenomenon of concentration of molecule of gas or liquid at solid surface is called as adsorption.
- This substance the concentration at the surface is called as adsorbate and the solid on whose surface the concentration occurs is called as adsorbent.

Absorbend

Adsorbent

- Adsorption occurs only the concentration of the solid surface is called as adsorption implies precaution into the body of the solid surface is mainly two phase.
- Physical adsorption
- Chemical adsorption.

Physical adsorption:

Is due to gas molecules being held to the solid surface by van der Waals's force.

Chemical adsorption:

Is due to the gas molecules held to the solid surface by chemical bond.

Procedure:

STANDARDISATION OF 0.1N NaOH:

- Weigh accurately 0.5 gm of KOH phthalate and dissolve in 50 ml of water. Then the solution titrated against 0.1N NaOH using appearance of pale pink colour.
- Each ml of 0.1 N NaOH is equivalent to 0.020242 gm of potassium hydroxide phthalate.

PROCEDURE FOR DETERMINATION OF % ADSORPTION OF OXALIC ACID BY CHARCOAL METHOD:

- Prepare 0.1 N oxalic acid freshly from the above oxalic acid solution pipette out 20 ml of solution in a clean conical flask and titrated against 0.1 N NaOH using phenolphthalein as indicator calculate the amount of oxalic acid present in 20 ml of solution.
- Pipette out 20 ml of oxalic acid and add 0.25 gm of activated charcoal.
- Then the solution is titrated with NaOH using phenolphthalein as an indicator.
- Calculate the amount of oxalic acid present in 20 ml of above solution.
- Each ml of 0.1 N NaOH is equivalent to 0.06604 gm of oxalic acid.

S.no	Contents of conical flask	Burette reading		Volume of 0.1 N NaOH (ml)	Indicator	End point
		Initial (ml)	Final (ml)			

Normality of 0.1 N NaOH = Weight taken X actual molarity titrate/value X equivalent weight factor

0.1 N Oxalic acid V_s 0.1N NaOH.

S.no	Contents of conical flask	Burette reading		Volume of 0.1 N NaOH (ml)	Indicator	End point
		Initial (ml)	Final (ml)			

The amount of oxalic acid before addition of charcoal

$$= \frac{\text{Titrate value X Strength of 0.1N NaOH X Equivalent weight factor}}{\text{Actual molarity}}$$

S.no	Contents of conical flask	Burette reading		Volume of 0.1 N NaOH (ml)	Indicator	End point
		Initial (ml)	Final (ml)			

The amount of oxalic acid in given sample after addition of charcoal.

The percentage adsorption of oxalic acid with charcoal.

$$\frac{C_1 - C_2}{C_1} * 100$$

Report:

- Amount of oxalic acid present before adding of activated charcoal = ----gm
- Amount of oxalic acid present after activated charcoal = ----- gm.
- The % adsorption 0.1 M oxalic acid add by charcoal was found to be = ----%

9. DETERMINATION OF CRITICAL SOLUTION TEMPERATURE OF PHENOL –WATER SYSTEM

Aim:

To determine the critical solution temperature of phenol –water system.

Requirements:

Chemicals:

- Phenol in liquid state.
- Distilled water.

Apparatus:

- Analytical balance
- Hard glass tube
- Thermometer
- Beakers
- Stirrer
- Water-bath

Principle:

- Critical solution temperature is defined as the temperature at which two conjugate solutions merge into one another to form one layer. This is characteristic of a particular system.
- When a small quantity of phenol is mixed with water and the mixture is shaken, phenol dissolves forming a single layer and adding layer quantities of phenol however two kinds of composition of liquid are formed. The lower layer consists of a small amount of water dissolved in phenol and the upper layer of phenol dissolved in water.

Procedure:

- Weigh accurately 4 gm of phenol in a glass tube.
- Introduce glass rods and thermometer into it, fix the tube in a vertical position in a water bath, set up the apparatus.
- Add 2ml of distilled water to phenol and keep the test tube in a water bath whose temperature is initially raised to about 30 ° C on a low flame with constant stirring. Note the temperature (T_1) of the solution obtained.
- Remove the tube from the water bath and allow it to cool. It forms a turbid solution as the temperature decreases. Note this temperature as (T_2). The mean of T_1 and T_2 is the transition temperature (T).
- Add 2ml more of distilled water to the same tube and determine the transition temperature as in the previous step. Continue the addition of 2ml distilled water at the same time to the same tube and record

the transition temperature each time till the maximum transition. Temperature is reached and comes down to about 40° C.

$$\text{percent by weight of phenol} = \frac{W_1}{W_1 + W_2} * 100$$

Weight of phenol (W_1) =

Weight of water (W_2) =

Density of distilled water =

Density of phenol =

Water added = Vml

S.no	Volume of water added (ml)	Weight of water ($w_2 = v \times d$)	Transition temperature (° C)		
			While heating(T_1)	While cooling (T_2)	Mean (T)

Report:

The critical solution temperature for phenol – water system is 40 C and its occurs at a concentration of 33 % percentage by weight of phenol.