

PHYSICAL PHARMACEUTICS- I

III SEMESTER (2nd YEAR B.PHARM)

PRACTICAL LAB MANUAL

Physical Pharmacy Laboratory Manual
(III rd. Semester)

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Expt.No.1

Determination of pK_a value by Half Neutralization/ Henderson Hasselbalch equation

REQUIREMENTS

Chemicals: 0.04N acetic acid, 0.04N NaOH (Standard), Buffer solution of known pH

Apparatus: Burette, Beakers, Stirrer, pH meter

PROCEDURE

Standardise the pH meter by the use of a buffer solution of known pH. Pipette out 25ml of 0.04N acetic acid solution in a dry beaker. Dip the electrode into the acid. Measure the pH. Add from the burette 1ml of 0.04N NaOH and stir. Measure the pH (do not remove the glass rod used for stirring). Continue addition of 1ml of NaOH till there is sudden change in pH and thereafter take at least three more readings. After each addition measure the pH.

Calculation

Read the end point of the titration from graph. Let it be x ml of 0.04N NaOH: Now from graph (1) read the pH for $V = x/2$, i.e. read the pH at half neutralization stage at which $[\text{Salt}] = [\text{Acid}]$ and from equation (1) $\text{pH} = \text{pK}_a$.

Therefore $K_a =$

Calculation of dB:

Since, 1000 ml of 1N NaOH = 1 equivalent of NaOH

Therefore 2 ml of 0.04 N NaOH = $(2 \times 1 \times 0.04) / (25 \times 1000)$ equivalent of NaOH

If 'x' ml of 0.04N NaOH is added to 25ml of 0.04N acetic acid solution, the total volume of the solution is (25+x) ml and gram equivalent of NaOH per litre of solution added per 2 ml increment in NaOH is,

$\text{dB} = (2 \times 1000) / [(25 \times 1000) (25+x)] = 2 / 25 \times \text{total volume}$

CONCLUSION

The buffer capacity (β_{max}) is _____

pK_a of acetic acid = _____ at room temperature

K_a for acetic acid = _____ at room temperature.

Expt.No.2

Partition co-efficient of benzoic acid in benzene and water

Aim: To determine the partition co-efficient of benzoic acid between benzene and water.

REQUIREMENTS

Chemicals: Saturated solution of benzoic acid in benzene, Benzene, 0.01N NaOH, 0.1N NaOH, Distilled water

Apparatus: Separating funnel: 250 ml, Conical flask, Pipette, Burette, Stoppered bottles

PROCEDURE

Prepare the following mixtures in separating funnels:

Set I: 25ml water + 25 ml of saturated solution of benzoic acid in benzene

Set II: 25 ml water + 20 ml saturated solution of benzoic acid in benzene + 5 ml benzene

Set III: 25ml water + 15 ml saturated solution of benzoic acid in benzene + 10 ml benzene

Shake the mixture in the separating funnel vigorously for about 30 minutes so that the benzoic acid gets distributed between the two solvents and the distribution equilibrium is reached. Allow the flasks to stand for 10 minutes to separate into two clear layers (remove the stopper of the separating funnel and keep its mouth open during this period to facilitate the separation). Drain off the lower aqueous layers in 3 different stoppered dry bottles. (Discard the intermediate layer between the two phases). Benzene layer remains in the separating funnels. Using a dry pipette take 5 ml of organic layer (Benzene) into a conical flask containing 10ml of water and titrate against 0.1 N NaOH using phenolphthalein as an indicator. End point: Colorless to pink. Pipette out 10 ml of the aqueous layer using a dry pipette and titrate it against 0.01 N NaOH solution using phenolphthalein as an indicator. End point will be colorless to pink.

Expt.No.3

Partition co-efficient of Iodine in Distilled water and Carbon tetrachloride

Aim: To establish the partition co-efficient of iodine in distilled water (d.w.) and carbon tetrachloride.

REQUIREMENTS

Chemicals: Distilled water, Iodine crystals, Carbon tetrachloride, 0.01N $\text{Na}_2\text{S}_2\text{O}_3$, 0.1N $\text{Na}_2\text{S}_2\text{O}_3$, Starch indicators

Apparatus: Separating funnels, Conical flask, Pipette, Burette, Reagent bottles

PROCEDURE

Approximately prepare 110 ml of saturated solution of iodine in carbon tetrachloride for use as stock solution.

Prepare the following mixtures in separating funnels:

Set I: 50 ml water + 40ml stock solution + 10 ml of CCl_4

Set II: 50 ml water + 30ml stock solution + 20 ml of CCl_4

Set III: 50 ml water + 20ml stock solution + 30 ml of CCl_4

Set IV: 50 ml water + 10ml stock solution + 40 ml of CCl_4

Shake the mixture in separating funnel forcefully for about 30 minutes for the iodine to get distributed between the two solvents and to reach the distribution equilibrium. Let the flasks to stand for about 10 minutes so that two clear layers are separated. Now remove stopper of the separating funnel and keep its mouth open during this period to facilitate the separation. Release the lower layer which is aqueous into 4 different stoppered dry bottles. (Discard the intermediate layer between both the phases). Organic layer stays in the separating funnels. Using a dry pipette withdraw 10 ml of organic layer to a conical flask. Titrate it against 0.1N $\text{Na}_2\text{S}_2\text{O}_3$. Starch is used as an indicator. End point: Blue color will disappear. Withdraw 10 ml of the aqueous layer using a dry pipette and titrate it against 0.01N $\text{Na}_2\text{S}_2\text{O}_3$ solution using starch as an indicator. End point: Blue color will disappear. Repeat the process for all the mixtures.

Expt.No.4**Determination of critical solution temperature (CST) of phenol-water system**

Aim: To determine the critical solution temperature of phenol-water system.

REQUIREMENTS

Chemicals: Phenol in liquid state

Apparatus: Hard glass tube, Thermometer, Beakers, Stirrer, Water bath

PROCEDURE

Weigh accurately 4 gms (w1 gm) of phenol in a glass tube. (Do not touch phenol, it is corrosive). Introduce glass rod and thermometer (with 0.1°C accuracy) into it. Fix the tube in a vertical position in a water bath (beaker containing water). Set up the apparatus as shown in the figure. Add 2 ml of distilled water to phenol and heat the test tube in a water bath, whose temperature is initially raised to about 30°C on low flame with constant stirring. Note the temperature (T1) of solution at which a clear and transparent solution is obtained. It is the solution of water in phenol. Remove the tube outside the water bath and with constant stirring note the transition temperature $t^{\circ}\text{C}$ at which the turbidity reappears (this is because water separates out as a different layer.). Add 2 ml more of distilled water and record the transition temperature. Continue the addition of 2 ml distilled water and record the transition temperature till the temperature goes through a maximum value (CST) and comes down to about 40°C.

Expt.No.5

Determination of surface tension by drop count method & drop weight method

Aim: To determine the surface tension of given liquid by drop count method & drop weight method using stalagmometer.

REQUIREMENTS

Apparatus: Stalagmometer, Beaker, Measuring cylinder

Chemicals: Distilled water, Ethanol, Acetone, Benzene

PROCEDURE

Drop count method

The liquid which is to be tested was sucked into the clean stalagmometer up-to mark A. Then it was allowed to fall down due to gravity. The number of drops were calculated when the liquid passes from mark A to B.

The procedure was repeated 3 times to obtain the mean value. Then the surface tension can be calculated using the formula.

$$v_1 = (n_2 \rho_1 / n_1 \rho_2) \times v_2$$

Where,

v_1 = surface tension of test liquid

v_2 = surface tension of water

ρ_1 & ρ_2 = Density of test liquid & water

n_1 = no. of drops of test liquid

n_2 = no. of drops of water

Drop weight method

The liquid whose surface tension is to be determined was sucked into the capillary tube then it is allowed to fall down due to gravity.

30 drops of liquid were collected in a beaker. The weight of the drops were determined by using electronic balance. The same procedure was repeated 3 times to obtain the mean value.

By using the value of w_1 & w_2 , the surface tension of liquid can be determined.

$$v_1 = (w_1 / w_2) \times v_2$$

Where,

v_1 = surface tension of test liquid

v_2 = surface tension of water

w_1 = weight of test liquid

w_2 = weight of water

Expt.No.6

Determination of HLB value of given surfactant

Aim: To determine the HLB value of a given surfactant

Griffin's method

Griffin's method for non-ionic surfactants as described in 1954 works as follows:

$$HLB = 20$$

Where S= Saponification number

$$HLB = 20 * M_h / M$$

$$HLB = 20 * M_h / M$$

where M_h

is the molecular mass of the hydrophilic portion of the molecule, and M is the molecular mass of the whole molecule, giving a result on a scale of 0 to 20. An HLB value of 0 corresponds to a completely lipophilic/hydrophobic molecule, and a value of 20 corresponds to a completely hydrophilic/lipophobic molecule.

REQUIREMENTS

Chemicals: Distilled water, Fatty acid ester e.g. Glyceryl monostearate (gm), 0.5N alcoholic potassium hydroxide (KOH), Stearic acid, ether, 0.5N hydrochloric acid, 0.1N sodium hydroxide, Phenolphthalein indicator.

Apparatus: Round bottom flask, Reflux condenser, Beakers, Burette, Pipette, Conical flask, etc.

PROCEDURE

1. Preparation of 0.5N alcoholic KOH

Dissolve around 4 g of KOH in 3 to 5 ml distilled water in a volumetric flask and make up to total volume to 100 ml with alcohol. Allow it to stand for about 24 hours and separate out clear liquid by decantation. Use this clear solution for experiment. Alcoholic KOH is used, because surfactants are freely soluble in alcohol than in water. The solubility improved by alcohol hydrolysis is effective one.

2. Determination of saponification number

Weigh accurately 0.5g of GMS and transfer into round bottom flask, add 15ml of alcoholic potassium hydroxide to it and reflux on boiling water bath for about half an hour.

Reflux separately 15 ml of alcoholic potassium hydroxide (without GMS) on boiling water bath for about an hour blank reading.

Cool both the solutions to room temperature and titrate separately against 0.5N hydrochloric acid using phenolphthalein as the indicator. (End point: pink to colorless or slightly yellowish)

Let the titre reading for sample (GMS) be V_1 and blank be as V_2 .

3. Determination of acid number

Weigh accurately 0.5g of stearic acid; add it to a mixture of 10ml of alcohol and 10ml of ether. If stearic acid does not dissolve in the solvent mixture, warm it on water bath until it dissolves. (Note: take care while warming, since both ether and alcohol are highly inflammable liquids)

Titrate solution of stearic acid against 0.1N sodium Hydroxide using phenolphthalein as the indicator.

Let the titre reading be V_3 .

Expt.No.7**Determination of Freundlich and Langmuir Constants using Activated charcoal**

Aim: To find out the monolayer capacity and specific surface area (area per gram) of given powder (activated charcoal) by adsorption method.

REQUIREMENTS

Chemicals: Distilled water, Activated charcoal, 0.5N Acetic acid, 0.2N Sodium hydroxide, Phenolphthalein

Apparatus: Beakers, Burette, Pipette, Stand, Conical flask, Funnels

PROCEDURE

Determine the exact normality (N) of given acetic acid solution by titrating 10ml against 0.2N NaOH solution using phenolphthalein indicator. (End point: Colourless to pink). Prepare following mixtures of acetic acid and distilled water in five separate dry bottles and keep them in a water bath at room temperature.

Add 1gm of the activated charcoal to each of the five different dry bottles and keep them in a water bath at room temperature. Swirl the bottles for 40 mins to attain adsorption equilibrium. Filter the solutions using dry filter paper into five different dry flasks and determine the effective normality of each filtered solution (N_2) by titrating 10ml against standard 0.2N NaOH solution, using phenolphthalein indicator. (End point : Colourless to pink)

Expt.No.8**Determination of critical micellar concentration of suitable surfactants****REQUIREMENTS**

Chemicals: Suitable surfactants, Distilled water

Apparatus: Stalagmo-meter, Analytical balance.

Principle: Critical micelle concentration is defined as the concentration of surfactant at which the micelle is starts to forming.

PROCEDURE

250 ml of stocks solution of surfactant SLS was prepared using distilled water having concentration of 1mg per solution from the stocks solution strength of surfactant were prepared using distilled water i.e. 5,10,15,20,25,30,35,40 mg /ppm. Now the surface tension of each of the strength solution was determined. A graph was plotted taking concentration on X- axis and surface tension on Y-axis.

Expt.No.9**Determination of stability constant and donor acceptor ratio of PABA-Caffeine complex by solubility method**

Aim: To find out the stability constant and donor acceptor ratio of PABA-Caffeine complex by solubility method.

REQUIREMENTS

Chemicals: Distilled water, p-Amino benzoic acid (PABA), Caffeine, Sodium hydroxide, Phenolphthalein indicator

Apparatus: Volumetric flask, Conical flask, Pipette, Burette

PROCEDURE

Prepare stock solutions of caffeine (0.1M) and sodium hydroxide solution (0.025N). Prepare different concentrations of caffeine as per the Table 1. Then add equal quantities of PABA samples to each flask containing caffeine solutions. Then shake all the flasks till equilibrium (approx. 30 minutes). Then filter the samples with Whattman filter paper. Then titrate 10 ml of the sample against 0.025N sodium hydroxide solution using phenolphthalein indicator. The data should be filled in the observation table.. Then draw the phase solubility PABA by taking concentration of caffeine on X axis and concentration of PABA on Y axis. Then calculate the Stoichiometric ratio and Complex stability constant.