

TECHNOCRATS

Lab Work Book of

Physical Pharmaceutics –II

(BP - 407 P)

Department of Pharmacy

Lab Manual of
Physical Pharmaceutics –II
(BP - 407 P)

Price : ₹ 110/-

Edition :

© Copyright Reserved

No part of this book can be reproduced or transmitted in any form or by any means, electronic or mechanical without the written permission of the publisher.

Disclaimer

Every possible effort has been made to bring out this book accurately to fulfill aspirations of all readers. The publisher and their associates do not make any warranty with respect to accuracy, completeness of the book and hence can not be held liable in any way for the loss or damage whatsoever.

Printed & Published by :



TECHNOCRATS
PUBLICATIONS

Arera Colony, Bhopal.

e-mail : technocratspublications@gmail.com



TECHNOCRATS
PUBLICATIONS

Lab Work Book
of
Physical Pharmaceutics –II
(BP-407 P)

(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 the limitations.
- 5. Leadership skills:** Understand and consider the human reaction to change, motivation issues, leadership and team-building when planning changes required for fulfillment of practice, professional and societal responsibilities. Assume participatory roles as responsible citizens or leadership roles when appropriate to facilitate improvement in health and 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 order of a reaction.
- CO2: Analyze the properties of powders.
- CO3: Determine the surface phenomena and surfactant properties.
- CO4: Construct adsorption isotherms.
- CO5: Calculation of adsorption isotherms.

Index

S. No.	List of Experiments	Page No.
1	To determine the particle size, particle size distribution by using sieving method	01
2	To determine the particle size distribution using microscopic methods.	04
3	To determine the bulk density, true density and porosity.	08
4	To determine the angle of repose and influence of lubricant on angle of repose.	11
5	To determine the viscosity of liquid using Ostwald's viscometer.	14
6	To determine the sedimentation volume with effect of different suspending agent.	17
7	To determine the sedimentation volume with effect of different concentration of single suspending agent	20
8	To determine the viscosity of semisolid by using Brookfield viscometer.	23
9	To determine the reaction rate constant of first order.	26
10	To determine the reaction rate constant of second order.	29
11	To determine the accelerated stability studies.	32

Experiment No:-1

AIM

Determination of particle size, particle size distribution by using sieving method.

REFERENCE

Gaud R S & Gupta G D , “Practical Physical Pharmacy”, 1st edition , Reprint 2008, CBS Publishers & Distributors, New Delhi, Page No 211-12

REQUIREMENT

Maize powder, starch, weighing machine, paper

THEORY

Particle size or granule size refer to diameter of particles and influence solubility, dissolution and bioavailability of various drugs.

Particle size distribution (PSD) is also known as grain distribution. PSD may define as relative amount of particles present in dispersed fluids. Sieving method is one of the oldest methods carried with dry or wet materials and sieve are usually agitated to expose all the particles to opening.

The PSD of a material can be important in understanding its physical and chemical properties. It affects the strength and load-bearing properties of rocks and soils. It affects the reactivity of solids participating in chemical reactions, and needs to be tightly controlled in many industrial products such as the manufacture of printer toner, cosmetics, and pharmaceutical products

PROCEDURE

1. Weigh a sample and record the starting sample weigh
2. Weigh and record the weight of each sieve and the bottom pan in a stack.
3. Place the sample on the top sieve of a stack of sieves and cover the top sieve with a flat cover
4. Shake the stack (keeping it vertical) for a specified length of time at a predetermined speed.
5. When the shaking is complete, reweigh and record the weight of each sieve and the bottom pan.

OBSERVATION TABLES

Sieve no.	Weight retained on seieve(gm)	% Weight retained
	Total weight=	

CALCULATIONS

.....

.....

.....

.....

RESULT

.....

.....

.....

.....

VIVA QUESTION

Q.-1. Define frequency curve distribution?

.....

.....

.....

.....

Q.-2. Write different method for particle size analysis?

.....

.....

.....

.....

Q.-3. How to calculate particle size distribution?

.....

.....

.....

.....

Q.-4. How particle size influence dissemination?

.....

.....

.....

.....

Experiment No:- 2

AIM

To determine the particle size distribution using microscopic methods.

REFERENCES

CVS Subrahmanyam, "Laboratory Manual of Physical Pharmaceutics", First Ed, 2002 vallabh prakashan, Page no 54.

THEORY

A powder is a dry, bulk solid composed of a large number of very fine particles that may flow freely when shaken or tilted. Powders are a special sub-class of granular materials, although the terms powder and granular are sometimes used to distinguish separate classes of material.

The particle-size distribution (PSD) of a powder, or granular material, or particles dispersed in fluid, is a list of values or a mathematical function that defines the relative amount, typically by mass, of particles present according to size.

The PSD of a material can be important in understanding its physical and chemical properties. It affects the strength and load-bearing properties of rocks and soils. It affects the reactivity of solids participating in chemical reactions, and needs to be tightly controlled in many industrial products such as the manufacture of printer toner, cosmetics, and pharmaceutical products.

APPARATUS AND CHEMICALS

Microscope, eye piece micrometer, stage micrometer, glass slides, cover slip.

PROCEDURE

A. Calibration of eye piece micrometer

1. Standard stage micrometer is used to calibrate the eye piece micrometer.
2. Places the stage micrometer on the stage of the microscope.
3. Position the object to the centre of the objective.
4. Initially focus at low power (10x)
5. The scale of the stage micrometer is seen (100 division)
6. Switch over to high power (45x). Care should be taken to avoid any damage to the stage micrometer.
7. Focus the object clearly.
8. Carefully replace eye-piece with eye piece micrometer. Now small lines of the numbered scale represent the ruling of eye piece.

9. If necessary rotate the eye piece in such a manner to bring two scales parallel to each other.
10. Select two points. One point on the left side where division of both scales coincide and another on the right side where the division coincide.
11. Count the number of small division x eye piece and big division y record them.
12. The calculation of calibration can be done as follows

$$\text{Eye piece division} = \frac{\text{No of stage micrometer division}}{\text{No of eye piece division}} \times \text{least count}$$

B. Mounting of the sample

1. Transfers a small portion of the given sample on to a clean slide.
2. Add one or two drops of liquid paraffin.
3. Disperse the sample uniformly with the help of a brush. The particles should be independent and their distribution should be uniform.
4. Place the cover slip carefully.
5. Wipe the excess liquid with a blotting paper.
6. Place the slide on stage of microscope.

C. Measurement of particle size

1. Focus the slide in low magnification (10x). Observe the presence of individual particle
2. Shift to high power (45x) and focus the slide.
3. Measure the size of each particle in the term of eye piece division. A total of 300 particles should be considered.
4. Tabulate the particle in terms of division of eye piece and the no of particle.
5. Multiply the no of eyepiece division by their calibrated in above.
6. Classify the diameter into size ranges.
7. Arrange the frequency of particles in terms of no distribution
8. Transform data in different ways as mentioned table
9. Draw the size distribution curves such as normal.
10. Report the result in diameter.

OBSERVATION**Data Generated For Size Distribution**

Size range	Mean size range	Log d	No of particle in each size	% frequency no of particles	Cumulative % frequency no distribution

Conversion of No Distribution to Weight Distribution

Mean size range d	No of particle in each size n	$n \times d$	$n \times d^2$	$n \times d^3$	Percent $n \times d^3$ undersize	Cumulative % frequency undersize

RESULT

Length no diameter =

Surface no diameter =

Volume no diameter =

Surface length diameter =

Volume surface diameter =

No distribution: Geometric mean =

Geometric variation =

Weight distribution: Geometric mean =

Geometric variation =

VIVA QUESTION

Q.-1. What is particle size?

.....

.....

.....

.....

Q.-2. What is significant of size distribution?

.....

.....

.....

.....

Q.-3. What is geometric mean?

.....

.....

.....

.....

Q.-4. Define powder?

.....

.....

.....

.....

Experiment no:-3

AIM

TO determination of bulk density, true density and porosity.

REFERENCE

Gaud R S & Gupta G D , “Practical Physical Pharmacy”, 1st edition , Reprint 2008, CBS Publishers & Distributors, New Delhi, Page No:-220,21,23

REQUIREMENT

Bulk density apparatus, measuring cylinder, sample.

THEORY

Bulk Density:-The bulk density of a powder is the ratio of the mass of an untapped powder sample and its volume including the contribution of the interparticulate void volume. Hence, the bulk density depends on both the density of powder particles and the spatial arrangement of particles in the powder bed.

True Density: The density of the particles that make up a powder or particulate solid, in contrast to bulk density, which measures the average density of a large volume of the powder in a specific medium.

POROSITY

Porosity or void fraction is a measure of the void (i.e. “empty”) spaces in a material, and is a fraction of the volume of voids over the total volume, between 0 and 1, or as a percentage between 0 and 100%.

PROCEDURE

1. Taken 100 gm powder sample and pass through sieve.
2. 20gm of powder sample was filled in 50ml capacity measuring cylinder
3. Measuring cylinder was fixed on bulk density apparatus.
4. Timer was set and started the apparatus.
5. Bulk volume after tapping was noted.

OBSERVATION TABLE

Weight of powder	Bulk volume of powder	No.of tapping

CALCULATION

$$\text{Bulk density} = \frac{\text{Mass of powder}}{\text{Bulk volume of powder}}$$

$$\text{True density} = \frac{\text{Weight of the tablet}}{\text{Volume of tablet}}$$

$$\text{Porosity} = \frac{\text{Bulk volume-true volume}}{\text{Bulk volume}}$$

OBSERVATION

.....

.....

.....

.....

RESULT

.....

.....

.....

.....

VIVA QUESTION

Q.-1. Define porosity?

.....

.....

.....

.....

Q.-2. define Tapped volume?

.....

.....

.....

.....

Q.-3. What is fluff density ?

.....

.....

.....

.....

Q.-4. What is the formula of porosity?

.....

.....

.....

.....

Q.-5. What is Carr's index?

.....

.....

.....

.....

Experiment no:- 4

AIM

To determine the angle of repose and influence of lubricant on angle of repose.

REFERENCE

Gaud R S & Gupta G D , “Practical Physical Pharmacy”, 1st edition , Reprint 2008, CBS Publishers & Distributors, New Delhi, Page No-212-13

REQUIREMENT

Powder sample, Magnesium Stearate, beaker, dollar scale

THEORY

The steepest angle at which a sloping surface formed of loose material is stable. Angle of repose of powder is important in determining good powder flow. Several methods can be used to measure angle of repose of powder. In this experiment, two different materials with different characteristics, which are taken from dried bulk and mixed with glidant.

PROCEDURE

1. 100g of powder is prepared.
2. The powder material is poured through a funnel to form a cone.
3. The tip of the funnel should be held close to the growing cone and slowly raised as the pile grows, to minimize the impact of falling particles.
4. Stop pouring the material when the pile reached a predetermined height or the base a predetermined width.
5. Rather than attempt to measure the angle of the resulting cone directly, the height is divided by half the width of the base of the cone.
6. The inverse tangent of this ratio is the angle of repose.
7. The experiment is repeated with sand material of different characteristics.

OBSERVATION TABLE

Sample	Height of powder	Width of powder	Angle of repose without lubricant	Angle of repose with lubricant

CALCULATION

.....

.....

.....

.....

RESULT

.....

.....

.....

.....

VIVA QUESTION

Q.-1. Define lubricant?

.....

.....

.....

.....

Q.-2. How to calculate angle of repose?

.....

.....

.....

.....

Q.-3. How lubricant influence angle of repose?

.....

.....

.....

.....

Q.-4. What is the range of angle of repose for excellent flow?

.....

.....

.....

.....

Q.-5. Give examples of lubricant?

.....

.....

.....

.....

.....

Experiment no:- 5

AIM

Determination of viscosity of liquid using Ostwald's viscometer.

REFERENCES

Gaud R.S.& Gupta G.D. "Practical Physical Pharmacy," CBS Publishers & distributors, New Delhi, First Edition 2001, Page no.55

REQUIREMENTS

Reagents: Glycerin or solution or simple syrup, Distilled water and washing solution.

Equipment: Ostwald's viscometer, pycnometer/density bottle, Beaker, Thermometer, Burette stand, Electronic balance, Stopwatch, Glassmaker and conical flask.

THEORY

Viscosity is a measurement of how resistant a fluid is to attempts to move through it. A fluid with a low viscosity is said to be "thin," while a high viscosity fluid is said to be "thick." It is easier to move through a low viscosity fluid (like water) than a high viscosity fluid (like honey).

PROCEDURE

1. Thoroughly clean the viscometer with a mixture of warm chromic acid and if necessary clean the viscometer with solvent.
2. After cleaning the viscometer dry it completely by passing current of air or dry in the oven.
3. Fix the viscometer on the burette stand vertically.
4. Determine the density by pycnometer.
5. Fill the purified water in the viscometer.
6. Report the time taken to flow water between two points.
7. Repeat the reading at least three times carefully and take average of these reading for calculation.
8. Remove purified water from the viscometer and rinse with the liquid under investigation for two to three times and then fill the liquid in the same way as purified water.
9. Further arrange the equipment properly and report the time taken to flow of liquid between two points A and B.
10. Take reading at least three and take average of these reading for the calculation.

OBSERVATION

Weight of empty pycnometer =gm

Weight of pycnometer with water =gm

Weight of pycnometer with testing liquid =gm

S. No.	Liquid	Time taken to flow of liquid between two points (sec.)				Average (a+b+c)/3 (Sec.)
		1(a)	2 (b)	3 (c)		
1	Water					
2	Liquid					

CALCULATION

Calculate density of liquid as ρ

$$= \frac{\text{Weight of water at room temperature } (w_2 - w_1)}{\text{Volume of pycnometer } (V)}$$

Calculate viscosity of liquid using the following formula

$$\frac{t_1 d_1}{t_2 d_2} \times \eta_1$$

Where,

η_1 Viscosity of liquid

η_2 Viscosity of purified water at temperature (... $^{\circ}\text{C}$).

d_2 density of purified water at temperature (... $^{\circ}\text{C}$).

d_1 density of liquid calculated

t_1 flow time of liquid between two fixed point

t_2 flow time of water between two fixed point

RESULT

Viscosity of given liquid at room temperature was found to be

.....

.....

.....

.....

VIVA QUESTION

Q.-1. What is viscosity?

.....

.....

.....

.....

Q.-2. Write factor affecting the viscosity?

.....

.....

.....

.....

Q.-3. Write formula for viscosity?

.....

.....

.....

.....

Q.-4. Define viscometer?

.....

.....

.....

.....

Experiment no:- 6

OBJECTIVE

Determine sedimentation volume with effect of different suspending agent.

REFERENCES

1. Subhramanyam C.V.S, "Pharmaceutical Engineering – Principles And Practices", vallabh prakashan first reprint edition 2005, page no:24

THEORY

A pharmaceutical suspension is a coarse dispersion in which insoluble solid particle are dispersed in a liquid medium.

Theory of sedimentation Stokes relation describes the sedimentation velocity of a particle in a suspension

$$V = \frac{2r^2 (d_1 - d_2) g}{9\eta}$$

Where

V = velocity of the sedimentation in cm/ sec

R = particle radius

D = particle diameter in cm

d_1 and d_2 = density of the particle

η = density of the medium in poises

PROCEDURE

1. Struck a graph paper on clean measuring cylinders with 25 equal divisions on it.
2. Prepare slurry of calcium carbonate and pour in measuring cylinder. Observe the rate of sedimentation for 10 min.
3. Then prepare slurry of different suspending agents in the same concentration as sodium alginate, starch & agar and repeat the procedure.
4. Calculate the sedimentation time.

OBSERVATION

S.No	Time:	Sodium alginate+calcium carbonate slurry	Starch+ calcium carbonate slurry	Agar+ calcium carbonate slurry
1	0-1 min	cm	Cm	Cm
2	1-2 min	cm	Cm	Cm
3	2-3 min	cm	Cm	Cm
4	3-4 min	cm	Cm	Cm
5	4-5 min	cm	Cm	Cm
6	5-6 min	cm	Cm	Cm
7	6-7 min	cm	Cm	Cm
8	7-8 min	cm	Cm	Cm
9	8-9 min	cm	Cm	Cm
10	9-10 min	cm	Cm	Cm

RESULT

The sedimentation volume of different suspending agents was found to be

.....

.....

.....

.....

VIVA QUESTION

Q.-1. Define suspending agent?

.....

.....

.....

.....

Q.-2. What is suspension?

.....

.....

.....

.....

Q.-3. What is sedimentation?

.....

.....

.....

.....

Q.-4. What is coarse dispersion?

.....

.....

.....

.....

Experiment no:- 7

OBJECTIVE

Determination sedimentation volume with effect of different concentration of single suspending agent

REFERENCES

1. Subhramanyam C.V.S., "Pharmaceutical Engineering – Principles And Practices", vallabh prakashan first reprint edition 2005, page no:28

THEORY

A pharmaceutical suspension is a coarse dispersion in which insoluble solid particle are dispersed in a liquid medium.

Theory of sedimentation Stokes relation describes the sedimentation velocity of a particle in a suspension

$$V = \frac{2r^2(d_1 - d_2)g}{9\eta}$$

Where

V = velocity of the sedimentation in cm/ sec

R = particle radius

D = particle diameter in cm

d_1 and d_2 = density of the particle

η = density of the medium in poises

PROCEDURE

1. Struck a graph paper on a clean measuring cylinder with 25 equal division on it
2. Prepare slurry of calcium carbonate and pour in measuring cylinder. Observe the rate of sedimentation for 10 min.
3. Then add sodium alginate 2% and 5% and repeat the procedure.
4. Calculate the sedimentation time.

OBSERVATION

S.No	Time:	Sodium alginate (0.0%) +calcium carbonate slurry	Sodium alginate (2%) +calcium carbonate slurry	Sodium alginate (5%) +calcium carbonate slurry
1	0-1 min	cm	cm	Cm
2	1-2 min	cm	cm	Cm
3	2-3 min	cm	cm	Cm
4	3-4 min	cm	cm	Cm
5	4-5 min	cm	cm	Cm
6	5-6 min	cm	cm	Cm
7	6-7 min	cm	cm	Cm
8	7-8 min	cm	cm	Cm
9	8-9 min	cm	cm	Cm
10	9-10 min	cm	cm	Cm

RESULT

.....

.....

.....

.....

VIVA QUESTION

Q.-1. What is sedimentation?

.....

.....

.....

.....

Q.-2. What are suspending agents?

.....

.....

.....

.....

Q.-3. What is velocity?

.....

.....

.....

.....

Q.-4. What is suspension?

.....

.....

.....

.....

Experiment no:- 8

OBJECTIVE

Determination of viscosity of semisolid by using Brookfield viscometer.

REFERENCES

1. Gaud R.S.& Gupta G.D. "Practical Physical Pharmacy," CBS Publishers & distributors, New Delhi, First Edition 2001, Page no.78

REQUIREMENTS

Reagents: Carbopol-940 or sodium alginate or sodium CMC, or other gel forming agents, distilled water and washing solution.

Glassware: Brookfield viscometer, beaker and Electric points.

THEORY

Classical Brookfield viscometers employ the principle of rotational viscometry - the torque required to turn an object, such as a spindle, in a fluid indicates the viscosity of the fluid.

Torque is applied through a calibrated spring to a disk or bob spindle immersed in test fluid and the spring deflection measures the viscous drag of the fluid against the spindle.

The amount of viscous drag is proportional to the amount of torque required to rotate the spindle, and thus to the viscosity of a Newtonian fluid.

In the case of non-Newtonian fluids, Brookfield viscosities measured under the same conditions (model, spindle, speed, temperature, and time of test, container, and any other sample preparation procedures that may affect the behavior of the fluid) can be compared.

The viscosity of a fluid is a measure of its resistance to gradual deformation by shear stress or tensile stress.

Principle of this method is based on the stoke's law, V

$$V = \frac{2\pi^2(\rho - \rho_0)g}{9\eta_0}$$

Where,

V = velocity of spherical ball moving in the liquid of viscosity η_0 ,

ρ and ρ_0 = are density of ball

r = radius of the ball

PROCEDURE

1. Prepare gel using gel forming agent about 250gm.
2. Put the gel for 24 h for homogenization.
3. Fill the gel in a beaker or gel holder.
4. Select the spindle on the basis of viscosity.
5. Set up the instrument with level of the base and attach with a constant electric supply.
6. Clean the instrument and attach the selected spindle to the viscometer.
7. Rotate the spindle in the gel till gel get find constant dial reading.
8. Repeat the experiment at least for three times.
9. If you want to study the effect of temperature on viscosity, maintain the temperature for 20min. and then determine the viscosity.

OBSERVATION

Temperature =⁰C

Amount of gel =gm

Spindle No. =

Speed of the spindle in the gel =

Reading dial average of three readings =

CALCULATION

Viscosity of the gel = Dial reading \times value with the spindle no. & speed (provided by manufacturer of instrument)

RESULT

Viscosity of prepared sample was found to be

.....

.....

.....

.....

VIVA QUESTION

Q.-1. Define viscosity?

.....

.....

.....

.....

Q.-2. Write factor affecting viscosity?

.....

.....

.....

.....

Q.-3. What is gel?

.....

.....

.....

.....

Q.-4. What is stokes law?

.....

.....

.....

.....

Experiment no:- 9

OBJECTIVE

Determination of reaction rate constant of first order.

REFERENCES

Mohanta G.P., And Manna P.k., "Physical Pharmacy Practical Text", Pharma book syndicate, Hydrabad, 2006, Page No. 85.

REQUIREMENTS

Colorimeter and suitable filter (green/yellow), Thermometer, Colorimeter cuvette, graduated pipettes, Magnetic stirrer, conical flask

THEORY

A reaction is said to be first order if the concentration of one reactant is dependent on the concentration of the reactant and the rate of reaction is proportional to the first power of the concentration of the reaction.

$A \leftrightarrow \text{product}$

a 0 initially

$a-x$ x after time t

Rate of reaction $\frac{dx}{dt} \propto (a-x)$

$$\frac{Dx}{dt} = K (a-x)$$

Where, K is the reaction rate constant.

$$K = 2.303/t \log a/a-x$$

In a first-order reaction, the reaction rate is directly proportional to the concentration of one of the reactants. First-order reactions often have the general form $A \rightarrow \text{products}$. The differential rate for a first-order reaction is as follows:

$$\text{rate} = -\Delta[A]$$

$$\Delta t = k[A]$$

PROCEDURE

1. Measure out 100 cm³ of the pH 7.5 buffer solution into a conical flask.
2. Put this on a magnetic stirrer/ electric hotplate and bring the temperature up to 70 ° C.
3. Add 0.10g of powdered aspirin to the buffer solution and stir the mixture gently until the aspirin is completely dissolved. No measurements should be made until all the solid has dissolved

4. Pipette 5 cm³ of iron (III) nitrate reagent directly into a colorimeter tube. Pipette 1 cm³ from the reaction mixture and empty it into the colorimeter tube. Mix well and measure the absorbance. The intensity of the colour and, therefore, the absorbance value depends on the concentration of the hydrolysis product 2-hydroxybenzoic acid.
5. Take 1 cm³ samples every 15 minutes for at least 2 hours and treat them as in step 4
6. Use the calibration graph produced in Determination of 2-hydroxybenzoic acid to calculate the concentration of 2-hydroxybenzoic acid in solution at each time interval.

OBSERVATION

The rate equation for the hydrolysis of Aspirin is complex, but at a given pH in dilute aqueous solution the equation reduces to the first - order equation:

where, $[A]$ is the concentration of aspirin k is the rate constant (its value depends on temperature and pH)

1. From the concentration of aspirin, calculate the concentration of aspirin remaining in solution.
2. Plot a graph of aspirin concentration, $[A]$, against time.
3. Measure the tangent at various point on the $[A]$ vs time graph. These give the rate of reaction at various aspirin concentrations.
4. Plot a graph of rate of reaction against aspirin concentration. The slope of this graph gives the value of the rate constant, k , under the reaction conditions used.
5. The half - life of a reaction is the time it takes for the concentration of a reactant to halve or the concentration of a product to double. For a first order reaction the half - life is a constant value.

RESULT

.....

.....

.....

.....

VIVA QUESTION

Q.-1. Define first order reaction?

.....

.....

.....

.....

Q.-2. What is rate of reaction?

.....

.....

.....

.....

Q.-3. What is hydrolysis?

.....

.....

.....

.....

Q.-4. What is half life?

.....

.....

.....

.....

Experiment no:-10

OBJECTIVE

Determination of reaction rate constant of second order.

REFERENCES

Mohanta G.P., And Manna P.k., "Physical Pharmacy Practical Text", Pharma book syndicate, Hyderabad, 2006, Page No.85.

REQUIREMENTS

Volumetric flask, ethyl acetate.

THEORY

The simplest kind of second-order reaction is one whose rate is proportional to the square of the concentration of one reactant. These generally have the form $2A \rightarrow \text{products}$. A second kind of second-order reaction has a reaction rate that is proportional to the product of the concentrations of two reactants. Such reactions generally have the form $A + B \rightarrow \text{products}$. An example of the former is a dimerization reaction, in which two smaller molecules, each called a monomer, combine to form a larger molecule.

The differential rate law for the simplest second-order reaction in which $2A \rightarrow \text{products}$ is as follows:

$$\text{rate} = -\frac{\Delta[A]}{\Delta t} = k[A]^2$$

Consequently, doubling the concentration of A quadruples the reaction rate. For the units of the reaction rate to be moles per liter per second (M/s), the units of a second-order rate constant must be the inverse ($\text{M}^{-1} \cdot \text{s}^{-1}$). Because the units of molarity are expressed as mol/L, the unit of the rate constant can also be written as $\text{L}(\text{mol} \cdot \text{s})$.

For the reaction $2A \rightarrow \text{products}$, the following integrated rate law describes the concentration of the reactant at a given time:

$$\frac{1}{[A]} = \frac{1}{[A]_0} + kt$$

Because above Equation has the form of an algebraic equation for a straight line, $y = mx + b$, with $y = 1/[A]$ and $b = 1/[A]_0$, a plot of $1/[A]$ versus t for a simple second-order reaction is a straight line with a slope of k and an intercept of $1/[A]_0$.

PROCEDURE

1. In a 250-ml volumetric flask, prepare a 0.04M solution of ethyl acetate.
2. Weigh the required amount of ethyl acetate into a tared weighing bottle containing water.
3. Adding the ethyl acetate (+0.001g) to the water prevents loss of the ester from evaporation.
4. Keep the weighing bottle tightly closed before washing the solution of the ester into the volumetric

flask and diluting to the mark.

5. Calculate the exact molarity. This solution should be prepared on the same day it is used, since appreciable hydrolysis can take place in a few days even in a neutral solution.
6. Calculate the number of millilitres of standard 0.1 M NaOH required making 250.0 ml NaOH with exactly the same molarity as the ethyl acetate solution. With a buret, deliver the required NaOH solution into a 250-ml volumetric flask and dilute to the mark.
7. Prior to making a run, immerse the solutions of the ester and the base, and the conductance cell, in the 25°C thermostat and allow them to come to thermal equilibrium. A flask for mixing should also be immersed.
8. A special flask (Daniels et al., 1970) containing the ester and base in two compartments is especially convenient.
9. Pipet a 25-ml aliquot of base into the inner cylinder and 50ml of ester solution into the region outside the cylinder. Replace in the thermostat until it reaches thermal equilibrium. Clamp the empty conductance cell in the thermostat.

OBSERVATION

.....

.....

.....

.....

RESULT

.....

.....

.....

.....

VIVA QUESTION

Q.-1. What is rate of reaction?

.....

.....

.....

.....

Q.-2. What is second order reaction?

.....

.....

.....

.....

Q.-3. What is reaction rate?

.....

.....

.....

.....

Q.-4. What are the factors which affect reaction rate?

.....

.....

.....

.....

Experiment no:- 11

OBJECTIVE

Accelerated stability studies.

REFERENCES

Mohanta G.P., And Manna P.k., “Physical Pharmacy Practical Text”, Pharma book syndicate, Hyderabad, 2006, Page No. 89.

REQUIREMENTS

Drug, thermometer, beaker

THEORY

Stability testing is termed as a complex process because of involvement of a variety of factors influencing the stability of a pharmaceutical product. These factors include stability of the active ingredient(s); interaction between active ingredients and excipients, manufacturing process followed, type of dosage form, container/closure system used for packaging and light, heat and moisture conditions encountered during shipment, storage and handling.

The purpose of stability testing is to provide evidence on how the quality of a FPP varies with time under the influence of a variety of environmental conditions such as temperature, humidity and light and to establish a shelf-life for the FPP, to determine the storage conditions and the in-use stability

PROCEDURE

1. Freshly prepared ointment & put into the container.
2. The concentrations of drug from formulation were determined in hour's interval.
3. The temperature should maintain in between the $25^{\circ}\text{C} \pm 2^{\circ}\text{C}$.
4. Repeat the procedure for 24 hrs.
5. The obtained concentration is filled in the given below table.

OBSERVATION

Stability study of ointment formulation at $25^{\circ}\text{C} \pm 2^{\circ}\text{C}$

Time (Hours)	Temperature	Concentration of drug($\mu\text{g/ml}$)
0	$25^{\circ}\text{C} \pm 2^{\circ}\text{C}$	
1	$25^{\circ}\text{C} \pm 2^{\circ}\text{C}$	
2	$25^{\circ}\text{C} \pm 2^{\circ}\text{C}$	
4	$25^{\circ}\text{C} \pm 2^{\circ}\text{C}$	
8	$25^{\circ}\text{C} \pm 2^{\circ}\text{C}$	
12	$25^{\circ}\text{C} \pm 2^{\circ}\text{C}$	
24	$25^{\circ}\text{C} \pm 2^{\circ}\text{C}$	

RESULT:

.....

.....

.....

.....

VIVA QUESTION

Q.-1. -What is stability study?

.....

.....

.....

.....

Q.-2. What is half life?

.....

.....

.....

.....

Q.-3. Write factor affecting stability?

.....

.....

.....

.....

Q.-4. What is Accelerated stability studies?

.....

.....

.....

.....