

SUBJECT

PHARMACEUTICS-I

B. PHARM, 1st YEAR, 1st SEMESTER

PRACTICAL LAB MANUAL

LABORATORY MANUAL OF PHARMACEUTICS**LIST OF EXPERIMENTS PHARMACEUTICS**

SL NO.	<u>LIST OF EXPERIMENTS PHARMACEUTICS</u>
1	To prepare and submit Simple Syrup IP.
2	Prepare and submit Ferrous Phosphate Syrup IP
3	Prepare and submit Piperazine Citrate Elixir
4	Prepare and submit Paracetamol Pediatric Elixir
5	Prepare and submit Strong Solution of Ammonium Acetate
6	Prepare and submit Cresol with Soap Solution IP.
7	Prepare and submit Calamine Lotion
8	Prepare and submit Turpentine Liniment
9	Prepare and submit Liquid Emulsion
10	Prepare and submit Throat paint I.P
11	To prepare and submit 20 ml. of aqueous Iodine solution I.P (Lugol's solution)
12	To prepare and submit Magnesium Hydroxide Mixture (Milk of Magnesia)
13	To prepare and submit Aluminium hydroxide suspension
14	Prepare and submit absorbable dusting powder
15	Prepare and submit Glycerogelatin base suppositories
16	Prepare and submit 20 gm of effervescent granules
17	Prepare and submit 20 gm of sulfur ointment.
18	Preparation of Face powder
19	Preparation And Submission of Body Powder/ Dusting Powder
20	Preparation And Submission of Tooth Powder
21	Preparation & Submission of Tooth Paste
22	Preparation And Submission of Hair Gel
23	Preparation And Submission of Shaving Cream

Experiment 1

Object: To prepare and submit Simple Syrup IP.

References:

L. Lachman, H. A. Lieberman and J.L. Kanig, The Theory and Practice of Industrial Pharmacy, 4th edition, 1991, Varghese Publishing House, Bombay.

Sanmathi B.S., Mehta K.M., Gupta A., Dispensing Pharmacy: A Practical Manual, 3rd Edition, Pharma Med Press.

Formula:

Ingredients	Quantity
Sucrose	66.7gm
Purified water	100 ml

Theory

Syrups are sweet, viscous, concentrated aqueous solutions of sucrose or other sugars. Medicated syrups contain a therapeutic or medicinal agent. They offer a pleasant means of administering disagreeable tasting drugs. Sucrose is commonly used in the preparation of syrups but other sugars can also be used like dextrose, sorbitol, glycerin and propylene glycol. Concentration of sucrose in sugar based syrup is important. A dilute solution of sucrose supports the growth of microorganisms whereas a saturated solution may lead to crystallization of a part of sucrose under condition of varying temperature. In simple syrup sucrose at the concentration of 85% is dissolved in water. These are sometimes used as a coating on to the surface of the tablets. If some therapeutic agent is present, then it is called as medicated syrup. Some syrups does not contain therapeutic agent, instead they consists of flavouring agents. These are called flavoured syrups and are generally used as vehicles. For the preparation of extemporaneous products, flavoured syrups are used as the vehicles. Flavoured syrups are made by infusing simple syrups

with flavouring agents during the cooking process. A wide variety of flavouring agents can be used, often in combination with each other, such as herbs (rosemary), spices (chipotle chilis, cardamom), or aromatics (orange peel, lemongrass, ginger). For instance, syrups aromatics is prepared by adding certain quantities of orange flavoring and cinnamon water to simple syrup.

Syrup IP is a 66.7% w/w solution of sucrose in purified water and Syrup USP consists of 85% w/v (corresponding to 64.74% w/w) solution of sucrose in purified water. Both the concentration gives stable syrups resistant to microbial growth. Method of preparation includes hot process, percolation (cold process), addition of medicating or flavorings liquid to syrup and agitation without heat.

Procedure:

1. All glassware were washed and dried.
2. Required quantity of chemicals were taken and weighed.
3. Sucrose was added in half the quantity of water and allowed to heat to dissolve it with continuous stirring.
4. Solution was allowed to cool and more purified water was added to make up the required weight.
5. Prepared formulation was packed in suitable container, labeled and submitted.

Use: It is used as additive in various formulations

Storage: Store in well closed container in a cool and dry place.

Result: Simple Syrup is prepared and submitted.

Experiment 2

Object: Prepare and submit Ferrous Phosphate Syrup IP

References:

Laboratory manual of pharmaceuticals by C.V.S. Subramanyam, J Thimmo shetty, G.I.

Prabhushankar first edition published by vallabh publication New Delhi

Jain N.K., Sharma S. N. A Textbook of Professional Pharmacy, 4th edition, Vallabh Prakashan,

Delhi

Formula:

Ingredients	Quantity
Iron fehlings	4.3 gm
Phosphoric acid	80 ml
Calcium carbonate	13.6 gm
Potassium bicarbonate	1.0 gm
Sodium phosphate	1.0gm
Cochineal	3.5 gm
Sucrose	700 gm
Orange flower water	50 ml
Purified water qs	1000 ml

Theory:

Syrups are sweet, viscous, concentrated aqueous solutions of sucrose or other sugars. Medicated syrups contain a therapeutic or medicinal agent. They offer a pleasant means of administering disagreeable tasting drugs. Sucrose is commonly used in the preparation of syrups but other sugars can also be used like dextrose, sorbitol, glycerin and propylene glycol. Concentration of sucrose in sugar based syrup is important. A dilute solution of sucrose supports the growth of microorganisms whereas a saturated solution may lead to crystallization of a part of sucrose under condition of varying temperature. In simple syrup sucrose at the concentration of 85% is dissolved in water.

Syrup IP is a 66.7% w/w solution of sucrose in purified water and Syrup USP consists of 85% w/v (corresponding to 64.74% w/w) solution of sucrose in purified water. Both the concentration give stable syrups resistant to microbial growth. Method of preparation includes hot process, percolation (cold process), addition of medicating or flavorings liquid to syrup and agitation without heat.

Ferrous Phosphate Syrup is a preparation containing iron along with electrolytes, calcium, potassium and sodium. these electrolytes overcome the deficiency, which is most common in anaemic condition.

Iron supplements are used to treat iron deficiency and iron-deficiency anemia, where requirements for iron are greater than the body's ability to supply iron such as in inflammatory states.

Since iron is essentially supplied orally, syrup is used as a major ingredient in this preparation. It acts as sweetening agent.

Procedure:

1. All glassware were washed and dried.
2. Required quantity of chemicals were taken and weighed.

3. Preparation of Medicinal Contents

- Phosphoric acid is diluted with water and divided into two portions
- To one portion of diluted phosphoric acid, iron is added and heated on water bath until iron dissolves
- Calcium carbonate, potassium bicarbonate and sodium phosphate are dissolved in second portion of diluted phosphoric acid in a beaker by stirring (carbon dioxide is

allowed to evolve)

- Both the solutions were dissolved and filtered to remove the impurities (iron carbide and carbon)

4. Preparation of Vehicle

- Coloring agent is extracted from cochineal by boiling it for 15 mins with water
- Sugar is added to the above colored decoction, and heating is continued until sugar completely dissolves
- The hot syrup containing coloring agent is cooled, strained, washed with water to produce a specified volume

5. Mixing of Both the Parts

- The colored syrup is mixed with mixture containing medicaments
- To the above mixture, orange flower water is added and the final volume is adjusted with water

6. The preparation was then transferred to light resistant container.

7. Container was labeled and submitted.

Use: Supplement of iron, sodium, calcium and phosphate

Dose: 2 to 8 ml

Storage: Store in well closed container in a cool place.

Result: Ferrous Phosphate Syrup is prepared and submitted.

Experiment 3

Object: Prepare and submit Piperazine Citrate Elixir

References:

Laboratory manual of pharmaceutics by C.V.S. Subramanyam, J Thimmo shetty, G.I.

Prabhushankar first edition published by vallabh publication New Delhi

Sanmathi B.S., Mehta K.M., Gupta A., Dispensing Pharmacy: A Practical Manual, 3rd Edition,

Pharma Med Press.

Formula:

Ingredients	Quantity
Piperazine citrate	180 gm
Chloroform spirit	5 ml
Glycerin	100 ml
Orange oil	0.25 ml
Syrup	500 ml
Purified water qs	1000 ml

Theory:

Elixirs are clear, sweetened, aromatic, hydro alcoholic liquid preparations intended for oral use.

They provide a palatable means of administering potent or nauseous drugs. Elixirs are less sweet and less viscous than syrups and may contain less or no sucrose, whereas elixirs are more stable.

Elixirs contain ethyl alcohol and suitable colorings and flavoring agent. Preservatives are not required as their alcohol content is sufficient to render them as self-preserving. They may also contain glycerin and syrup either for increasing the solubility of medicament or for sweetening purpose. These are stable preparation when packed in airtight, light resistant containers and if these are not diluted or mixed with other preparations.

Piperazine Citrate Elixir is used as anthelmintics, these are group of anti-parasitic drugs that expel parasitic worms and other internal parasites from the body by either stunning or killing them and without causing significant damage to the host.

Glycerin acts as co-solvents to enhance the solubility of the drug. Chloroform spirit acts as preservative. Orange oil acts as flavoring agents.

Piperazine citrate possesses unpleasant taste, so simple syrup is used to mask the taste of the drug. Piperazine Citrate Elixir is an oral solution containing 18.75% w/v of Piperazine Citrate in a suitable flavoured vehicle.

Procedure:

1. All glassware were washed and dried.
2. Required quantity of chemicals were taken and weighed.
3. Piperazine citrate was dissolved in small quantity of water.
4. To the solution, required quantity of orange oil, chloroform spirit, glycerin and syrup were added gradually with constant stirring.
5. To the prepared solution purified water was added to make up the required volume.
6. Prepared formulation was packed in suitable container, labeled and submitted.
7. The preparation was then transferred to light resistant container.
8. Container was labeled and submitted.

Use: Anthelmintic

Dose: 4 to 15 ml

Storage: Store in well closed container in a cool place.

Result: Piperazine Citrate Elixir is Prepared and submitted.

Experiment 4

Object: Prepare and submit Paracetamol Pediatric Elixir

References:

Laboratory manual of pharmaceuticals by C.V.S. Subramanyam, J Thimmappa Shetty, G.I.

Prabhushankar first edition published by Vallabh Publication New Delhi

Sanmathi B.S., Mehta K.M., Gupta A., Dispensing Pharmacy: A Practical Manual, 3rd Edition,

Pharma Med Press.

Formula:

Ingredients	Quantity
Paracetamol	24 gm
Amaranth solution	2 ml
Chloroform spirit	20 ml
Conc raspberry juice	25 ml
Alcohol 95%	100 ml
Propylene glycol	100 ml
Invert syrup	275 ml
Glycerin	1000 ml

Theory:

Elixirs are clear, sweetened, aromatic, hydro alcoholic liquid preparations intended for oral use. They provide a palatable means of administering potent or nauseous drugs. Elixirs are less sweet and less viscous than syrups and may contain less or no sucrose, whereas elixirs are more stable. Elixirs contain ethyl alcohol and suitable colorings and flavoring agent. Preservatives are not required as their alcohol content is sufficient to render them as self-preserving. They may also contain glycerin and syrup either for increasing the solubility of medicament or for sweetening purpose. These are stable preparation when packed in airtight, light resistant containers and if these are not diluted or mixed with other preparations.

Glycerin acts as co-solvents to enhance the solubility of the drug. Chloroform spirit acts as preservative. Concentrated raspberry juice acts as flavoring agents.

Pediatric paracetamol elixir improves the patient's condition by increasing the pain threshold and increases the blood flow across the skin, heat loss and sweating. Headache and toothache are among the most common reported uses for pediatric paracetamol elixir.

Pediatric paracetamol elixir (paracetamol elixir) contains paracetamol to relieve pain and reduce high temperatures. Paracetamol elixir can be used in babies and children for the treatment of mild or moderate pain and feverishness, and also in babies who develop fever after vaccination. Before giving paracetamol elixir to child do not give paracetamol elixir if the child is taking any other paracetamol containing products, other flu, cold, cough or decongestant products, or alcohol. Do not give paracetamol elixir to child if he/she is allergic (hypersensitive) to paracetamol, or any of the other ingredients of paracetamol elixir or if child has kidney or liver

Procedure:

1. All glassware were washed and dried.
2. Required quantity of chemicals were taken and weighed.
3. Alcohol, propylene glycol, chloroform spirit and Conc raspberry juice were added
4. Paracetamol was taken into beaker and the above mixture was added slowly to dissolve paracetamol completely
5. To above mixture, invert sugar was added.
6. Amaranth solution was added and glycerin was added to make up the required volume
7. Solution was filtered if necessary
8. The preparation was then transferred to light resistant container.
9. Container was labeled and submitted.

Use: analgesic and antipyretic for children

Dose: 5 to 10 ml

Storage: Store in well closed container in a cool place.

Result: Paracetamol Paediatric Elixir is Prepared and submitted.

Experiment 5

Object: Prepare and submit Strong Solution of Ammonium Acetate

References:

Laboratory manual of pharmaceutics by C.V.S. Subramanyam, J Thimmo shetty, G.I.

Prabhushankar first edition published by vallabh publication New Delhi

Sanmathi B.S., Mehta K.M., Gupta A., Dispensing Pharmacy: A Practical Manual, 3rd Edition,

Pharma Med Press.

Formula:

Ingredients	Quantity
Glacial acetic acid	453 gm
Ammonium bicarbonate	470 gm
Ammonia solution strong	100 ml
Purified water q.s.	1000 ml

Theory:

A solution is a homogeneous mixture composed of two or more substances. In such a mixture, a solute is dissolved in another substance, known as a solvent. A common example is a solid, such as salt or sugar, dissolved in water, a gases may dissolve in liquids, for example, carbon dioxide or oxygen in water. Liquids may dissolve in other liquids. Gases can combine with other gases to form mixtures, rather than solutions. All solutions are characterized by interactions between the solvent phase and solute molecules or ions that result in a net decrease in free energy. Under such a definition, gases typically cannot function as solvents, since in the gas phase interactions between molecules are minimal due to the large distances between the molecules. This lack of interaction is the reason gases can expand freely and the presence of these interactions is the reason liquids do not expand. Solutions should be distinguished from non-homogeneous mixtures such as colloids and suspension.

Solutions are liquid preparations containing one or more chemical substances usually dissolved in water. Solutions are used for specific therapeutic effect of solute either internally or externally. It contains 57.5% w/v of ammonium acetate. In this preparation two alkaline substances (ammonium bicarbonate and ammonia solution strong) are used because it is not possible to prepare the solution by reacting glacial acetic acid with ammonium bicarbonate alone because at certain point the reaction between these two substance ceases and the desired product is not produced so ammonia solution strong is used to complete neutralization of the acid and to make the preparation alkaline having pH between 7.6 to 8.1. As the salt of a weak acid and a weak base, ammonium acetate is often used with acetic acid to create a buffer solution.

Procedure:

1. All glassware were washed and dried.
2. Required quantity of chemicals were taken and weighed.
3. Glacial acetic acid was mixed with about 350ml of purified water, to this ammonium bicarbonate was added in small quantities with continuous stirring until completely dissolved.
4. Then small quantity of ammonia solution was added until one drop of the resulting solution diluted with 10 drops of water, gives full blue color with 1 drop of bromothymol blue solution and a full yellow color with one drop of thymol blue solution.
5. Then sufficient amount of purified water was added to produce required volume.
6. Prepared formulation was packed in suitable container, labeled and submitted.

Use: It is diaphoretic, which are used to lower the raised body temperature by increasing the excretion of body fluids in the form of sweat and urine

Dose: 1 to 4 ml

Result: Strong ammonium acetate solution is Prepared and submitted.

Experiment 6

Object: Prepare and submit Cresol with Soap Solution IP.

References:

Laboratory manual of pharmaceutics by C.V.S. Subramanyam, J Thimmo shetty, G.I.

Prabhushankar first edition published by vallabh publication New Delhi

Sanmathi B.S., Mehta K.M., Gupta A., Dispensing Pharmacy: A Practical Manual, 3rd Edition,

Pharma Med Press.

Formula:

Ingredients	Quantity
Cresol	500 ml
Vegetable oil	180 gm
Potassium hydroxide	42 gm
Purified water	1000 ml

Theory:

Cresol is soluble in water to the extent of 2% v/v. But Lysol (cresol with soap) containing 50% v/v of cresol is very effective disinfectant. It kills microorganisms, as it possesses bactericidal and detergent properties. Solubility of cresol in water can be enhanced using soap.

It acts by disruption of cell membranes and denaturation of proteins and enzymes of the cell. It is effective against vegetative gram positive and gram negative bacteria, mycobacterium and viruses.

Soaps are surfactants, which form micelles above critical micelle concentration. At this stage cresol gets selectively entrapped inside spherical micelles. Thus, solubility of cresol is increased.

The soap is prepared by a saponification reaction between alkali and vegetable oils (or fatty acids). The vegetable oil may be cottonseed, linseed, soyabean or similar oils (excluding coconut and palm kernel oils). The alkali is potassium hydroxide solution. Alternatively, sodium hydroxide solution may be used.

Test to check completion of saponification reaction is to add few drops of water to the reaction mixture, if it remains completely miscible means the reaction is completed and if the mixture remains immiscible, it shows reactions is incomplete and heating should be continued for completion of saponification reaction.

Cresol with soap solution cannot be used on human beings (as an antiseptic) because of its necrotic action to animal tissues. Even 5 to 10% aqueous solution of cresol irritate the skin of many people.

Procedure:

1. All glassware were washed and dried.
2. Required quantity of chemicals were taken and weighed.
3. Potassium hydroxide is dissolved in small quantity of purified water.
4. Vegetable oil is added to the above alkali solution.
5. The above mixture is heated on water bath with continuous mixing
6. The solution was heated continuously until the completion of saponification reaction.
7. Cresol was added and mixed thoroughly
8. Sufficient water was added to produce the required volume.
9. The preparation was then transferred to light resistant container.
10. Container was labeled and submitted.

Use: Disinfectant

Storage: Store in well closed container in a cool place.

Result: Cresol with soap solution is Prepared and submitted.

Experiment 7

Object: Prepare and submit Calamine Lotion

References:

Laboratory manual of pharmaceuticals by C.V.S. Subramanyam, J Thimmo shetty, G.I. Prabhushankar first edition published by vallabh publication New Delhi
Sanmathi B.S., Mehta K.M., Gupta A., Dispensing Pharmacy: A Practical Manual, 3rd Edition, Pharma Med Press.

Formula:

Ingredients	Quantity
Calamine	150 gm
zinc oxide	50 gm
Bentonite	30 gm
Sodium citrate	5 gm
Liquefied phenol	5 ml
Glycerin	50 ml
Rose water, q.s	1000 ml

Theory:

A pharmaceutical suspension is a type of disperse system in which one substance, the insoluble solid (the disperse phase) is distributed throughout a vehicle (the continuous phase) along with other additives in the formulation (suspending agent, preservative, buffering system , coloring agent , flavoring agent and sweating agent).

Calamine is colored zinc carbonate (pink color) is practically insoluble in water, as zinc oxide. Both are astringents and indiffusible solids. Bentonite is the suspending agent used as thickening agent. Sodium citrate is added to control flocculation of calamine, by causing partial deflocculation of calamine, in its absence the suspension is much thicker and very difficult to pour from the bottle. Glycerin is used to thicken the product and help powder adherence to the skin. Liquefied phenol acts as a preservative and antiseptic.

Pharmaceutical suspensions **are** prepared for oral, external, parental, ophthalmic and inhalation use.

Calamine lotion, is a suspension used externally as cooling, anti pruritic preparation in case of sun burns, small pox etc. The color of calamine lotion is pink.

The most important auxiliary label is "for external use"," shake well before use" don't apply to broken skin the shelf life is one month. The storage condition is " store in cool not below 4C and dry place". The lotion should be applied to the affected areas when required and allowed to dry.

Procedure:

1. All glassware were washed and dried.
2. Required quantity of chemicals were taken and weighed.
3. Weigh and mix the calamine, zinc oxide and bentonite in a mortar so that the bentonite is well distributed.
4. - Dissolve sodium citrate in 700 ml rosewater, and gradually add to the mixture in the mortar, so that a smooth paste is produced
5. Add the liquefied phenol and glycerin and mix well
6. Add sufficient rose water to produce the required volume.
7. The preparation was then transferred to light resistant container.
8. Container was labeled and submitted.

Use: Calamine lotion is a suspension used externally as cooling, anti pruritic preparation in case of sun burns, itching and skin irritation etc

Storage: Store in well closed container in a cool place.

Result: Calamine Lotion is Prepared and submitted.

Experiment 8

Object: Prepare and submit Turpentine Liniment

References:

Laboratory manual of pharmaceutics by C.V.S. Subramanyam, J Thimmo shetty, G.I.

Prabhushankar first edition published by vallabh publication New Delhi

Sanmathi B.S., Mehta K.M., Gupta A., Dispensing Pharmacy: A Practical Manual, 3rd Edition,

Pharma Med Press.

Formula:

Ingredients	Quantity
Soft soap	90 gm
Camphor	50 gm
Turpentine oil	650 ml
Purified water	1000 ml

Theory:

Liniment (liquid or ointment) - dosage form for external use, is a fat liquid or gelatinous mass, which melt at body temperature. Wide application of liniment in medical practice due to their advantages: Medicinal substances of the liniment are well absorbed by the skin, and have high bioavailability and Compared with ointments liniments better applied to the skin, leaving fewer traces on skin and clothing of the patient. Liniment is prepared by the general rules of preparation of liquid dosage forms.

Turpentine Liniment is used as counter irritant and rubefacient. Camphor is soluble in turpentine oil but not in water. Turpentine oil is immiscible in water. therefore oil in water emulsion is formed using soft soap as emulsifying agent.

Procedure:

1. All glassware were washed and dried.
2. Required quantity of chemicals were taken and weighed.
3. Soft soap is weighed and transferred to a mortar and gradually mixed with small quantity of distilled water with constant trituration to get a cream-like consistency
4. Camphor was dissolved in turpentine oil by continuous stirring.
5. Camphorated turpentine oil is added in drops to the mortar with thorough trituration till whole of camphorated turpentine oil is added.
6. The contents are transferred to a measuring cylinder and the required volume is made up by adding subsequent washings of a mortar and pestle with distilled water.
7. The liniment is thoroughly mixed.
8. The preparation was then transferred to light resistant container.
9. Container was labeled and submitted.

Use: Counter Irritant, Rubefacient

Storage: Store in well closed container in a cool place.

Result: Turpentine Liniment is Prepared and submitted.

Experiment 9

Object: Prepare and submit Liquid Emulsion

References:

Laboratory manual of pharmaceuticals by C.V.S. Subramanyam, J Thimmo shetty, G.I.

Prabhushankar first edition published by vallabh publication New Delhi

Sanmathi B.S., Mehta K.M., Gupta A., Dispensing Pharmacy: A Practical Manual, 3rd Edition,

Pharma Med Press.

Formula:

Ingredients	Quantity
Liquid paraffin	10 ml
Castor oil	10 ml
Simple syrup	30 ml
Water qs	100 ml

Theory:

An emulsion is essentially a liquid preparation containing a mixture of oil and water that is rendered homogeneous by the addition of an emulsifying agent. The emulsifying agent ensures that the oil phase is finely dispersed throughout the water as minute globules. This type of emulsion is termed an 'oil in water' emulsion. The oily phase (disperse phase) is dispersed through the aqueous phase (continuous phase). Generally all oral dose emulsions tend to be oil-in-water as the oily phase is usually less pleasant to take and more difficult to flavour. 'Water-in-oil' emulsions can be formed but these tend to be those with external uses. The pharmaceutical term 'emulsion' is solely used to describe preparations intended for internal use, i.e. via the oral route of administration. Emulsion formulations for external use are always given a different title that reflects their use, e.g. application, lotion and cream. Oral emulsions are oral liquids containing one or more active ingredients. They are stabilized oil-in-water dispersions, either or both phases of which may contain dissolved solids. Solids may also be suspended in oral emulsions. When issued for use, oral emulsions should be supplied in wide-mouthed bottles.

Stability of emulsions

Emulsions can break down in the following ways:

- cracking
- creaming
- phase inversion.

Liquid paraffin is a mineral oil obtained from petroleum. it gets emulsified in the GI tract and holds water. So the faecal matter do not become dry. Castor oil is a fixed oil obtained from castor seeds of *Ricinus Communis*. it produces the purgative effect. Liquid paraffin is a type of medicine called a laxative. It works by softening and lubricating the stools. This helps the stools to move more easily through the bowel. This medicine relieves constipation, making stools easier to pass.

Procedure:

1. All glassware were washed and dried.
2. Required quantity of chemicals were taken and weighed.
3. Castor oil and liquid paraffin are transferred into dry mortar, and triturate well.
4. Acacia is placed on the oil, triturated gently
5. When the gum is dispersed, water is added with rapid trituration.
6. Measured volume of syrup is diluted with water. This is gradually added to mortar with continuous trituration.
7. The content was transferred to measuring cylinder
8. Finally the volume was made up to the required level with water.
9. The preparation was then transferred to light resistant container.
10. Container was labeled and submitted.

Use: Laxative

Storage: Store in well closed container in a cool place. With a label "Shaken well before Use"

Result: Liquid Emulsion is Prepared and submitted.

Experiment 10

Object: Prepare and submit Throat paint I.P

References:

Laboratory manual of pharmaceuticals by C.V.S. Subramanyam, J Thimmo shetty, G.I.

Prabhushankar first edition published by vallabh publication New Delhi

Sanmathi B.S., Mehta K.M., Gupta A., Dispensing Pharmacy: A Practical Manual, 3rd Edition,

Pharma Med Press.

Formula:

Ingredients	Quantity
KI	25.0 gm
Iodine	12.5 gm
Ethanol	40 ml
Water	25 ml
Peppermint oil	4.0 ml
Glycerin	1000

Theory:

Throat Paints are solutions or dispersions of one or more active ingredients intended for application to the mucosa of the throat or mouth. Throat paints are viscous due to a high content of glycerin, which being sticky, adhere to the affected site and prolong the action of the medicaments.

Procedure:

- (i) Potassium iodide is dissolved in water.
- (ii) Iodine is added in the concentrated potassium iodide solutions to form KI_3 (or higher iodides).
- (iii) Peppermint oil is dissolved in alcohol 90% v/v and the alcoholic solution is added to the iodine solution.
- (iv) Volume is made up with glycerin.

ROLE OF INGREDIENT

- ❖ **Potassium Iodide:** To make soluble iodine in water
- ❖ **Iodine:** Antiseptic, Penetrate into pores and have germicidal effect, treat small abrasion and

wounds in Skin

❖ **Alcohol:** Preservative

❖ **Water:** Solvent

❖ **Peppermint Oil:** Flavoring agent

❖ **Glycerin:** Vehicle, Viscous, sticky, adhere to affected site and prolong effect of medicament

Use: used for pharyngitis or tonsillitis. Iodine throat paint is designed to kill germs. It can be used on sore throats and ulcers to ease them

Method of use:

❖ Apply with the help of soft brush or a cotton swab.

❖ Food and water before and after application of throat paint, should be avoided for 1 hr

Storage: A wide mouthed, fluted, light resistant, screw-capped, glass-jar is used.

Result: Throat Paint is Prepared and submitted.

Experiment 11

AIM: To prepare and submit 20 ml. of aqueous Iodine solution I.P (Lugol's solution)

Formula:

S.no	Ingredient	Quantity
1.	Iodine	5gm
2.	K I	10gm
3.	Purified water	100 ml.

Theory: Aqueous Iodine solution is supplement of KI inn the Iodine in different condition like goiter the patient iodine supplied in the solution from poly iodine complications.

Poly Iodine is easily soluble in water by ion induce dipolar interaction higher poly iodine higher poly iodine are concentration of the solution thefore I & KI are dissolve in the small quantity easily diluted to required volume is making iodine solution KI may replace by NaI.

Since iodine is react with some intrusting in ordinary in last contain. A resistant container like amber color used to stored`

Procedure:

1. Weight of quantity KI that is dissolve in a small quantity of purified water.
2. Weight quantity of iodine dissolves in the above solution.
3. Sufficient preformed water added to required volume. The preparation is transferred into well closed container in amber color.
4. The bottle is capped labeled published and submitted.

Category: Iodine supplement is iodine differential condition like goiter.

Dosage: .3-1 ml.

Storage: Store in a tightly closed container in a cool place.

Precaution: Iodine preparation should not administer during pregnancy & lactation this interfere with test for typhoid function some patient so keep.

Result : The aqueous Iodine solution has been prepared and submitted

Experiment 12

Aim: To prepare and submit Magnesium Hydroxide Mixture (Milk of Magnesia)

The following formula:-

Ingredients	Quantity
Magnesium sulfate	48.0 gm
Sodium hydroxide	15 gm
Light magnesium oxide	53 ml
Chloroform	3 ml
Purified water	1000 ml

Theory: Magnesium Hydroxide Mixture is an aqueous suspension of hydrated magnesium oxide. It may be prepared from a suitable grade of Light Magnesium Oxide.

Content of hydrated magnesium oxide, calculated as Mg(OH)_2 : 7.45 to 8.35% w/w.

Extemporaneous preparation

Procedure:-

Dissolve the sodium hydroxide in 150 ml of purified water, add the light magnesium oxide, mix to form a smooth cream and then add sufficient purified water. Pour this suspension in a thin stream into a solution of the magnesium sulfate in purified water, stirring continuously during the mixing. Allow the precipitate to subside, remove the clear liquid, transfer the residue to a calico strainer, and allow draining and washing the precipitate with purified water until the washings give only a slight reaction for sulfate. Mix the washed precipitate with purified water, dissolve the chloroform in the mixture and add sufficient purified water to produce 1000 ml.

Use: As an Antacid

Result: Magnesium Hydroxide Mixture is Prepared and submitted.

Experiment 13

Aim: To prepare and submit Aluminium hydroxide suspension

Formula:-

Ingredients	Quantity
Aluminium Hydroxide gel	36 gm
Sorbitol	7 gm
Methylparaben	200 mg
Propylparaben	200 mg
peppermint oil	.005 ml
Alcohol	1.00 ml
sufficient water, sufficient to produce	100 ml

Theory: - Aluminium Hydroxide gel is a aqueous suspension of hydrated aluminium oxide together with varying quantities of basic Aluminium carbonate and bicarbonate. It contains not less than 3.5% w/w and not more than 4.4% w/w of aluminium oxide. It may contain glycerin, sorbitol, sucrose or saccharin as sweetening agent, peppermint oil or other suitable flavours. It may also contain suitable antimicrobial agents. Aluminium hydroxide gel powder is one of the antacids used in pharmaceutical preparation. It is an insoluble antacid and is very reactive (neutralizing capacity) and strongly anti proteolytic. Aluminium hydroxide gel is either used in powder or paste form, depending upon the final product (i.e.) tablet or suspension. It is used externally as a mild astringent and desiccant and internally as antacid and protective. Since it is used as a drug item, special care is taken during processing to avoid any contamination and precautions are taken for standardization to IP grade. Aluminium hydroxide mixture is used to provide symptomatic relief in gastric and ulcer and in reflux oesophagitis and is used in the treatment of hyperchlorhydria. It may be administered in doses of 7.5 to 15 ml every 2 to 4 hours or more frequently in water or milk or tablets containing the dried ingredients may be sucked or chewed.

Procedure:-

1. Dissolve methylparaben, propylparaben, saccharin, peppermint oil in alcohol and purified water
2. Dissolve aluminium hydroxide gel and mix uniformly
3. Add sorbitol or mannitol and study the effect of conc of these substances and their conc. in the suspension

Category:- Antacid

Dose:- 7.5-15ml

Storage:- Preserve in a well –closed container

Result: Aluminium Hydroxide Solution is prepared and submitted.

Experiment 14

AIM:- Prepare and submit absorbable dusting powder

Equipments required:

Mortar and pestle, 90- mesh sieve, weighing balance

Formula:-

Ingredients	Quantity
Corn starch	98 gm
Light magnesium oxide	2 gm

Send 20 gm

Theory:- Dusting Powders is used a medicated or non-medicated for external application for various parts of the body as lubricants, protectives, absorbents, antiseptics, astringents and antiperspirants agents. Dusting Powders are usually dispensed in sifter containers for convenient application to the skin. Foot powders and talc powders are currently available as aerosols.

Procedure:-

- 1- Pulverize starch and magnesium oxide to fine powders, and pass through a 90-mesh sieve.
- 2- Triturate the two powders (starch over magnesium oxide) in a mortar with pestle method

Calculation :

Because of mechanical losses during preparation, you have to calculate for 25% excess so , instead of preparing 20 g , you will prepare 25 g , the total amounts in the prescription is 100 g , so, multiply each ingredient by a factor of (25/100).

Use : As lubricant for surgical gloves.

Result: Absorbable Dusting Powder is prepared and submitted.

Experiment 15

Aim:- Prepare and submit Glycero-gelatin base suppositories

Formula:

Ingredients	Quantity
Gelatin	14 g
Glycerin	70 g
Water QS	100g

Equipments Required:- Mortar and pestle, Porcelain crucible, water-bath, 2 g x 6 mold

Chemicals: Gelatin 84g, Glycerin 420 g, water 96 ml.

Theory:

A suppository is a solid or semisolid mass intended to be inserted into a body orifice (e.g. the rectum, Vagina, Urethra) to provide either a local or systemic therapeutic effect. Once inserted, a suppository either melts at body temperature or dissolves (or disintegrates) into aqueous secretions of the cavity.

Rectal suppositories are useful when oral administration is inappropriate, as with infants, debilitated individuals and patients with nausea, vomiting and gastrointestinal disturbances. Some drugs may cause irritation to the G.I.T. tract.

Gelatin dissolves in hot water, forming a solution which sets to a jelly. This fact is used to convert glycerin into solid form for use as suppository. A suitable gelatin jelly is also used as a base for other medicaments besides glycerin. A gelatin base is incompatible with many of the substances prescribed in suppositories, e.g. tannic acid, ferric chloride, Gallic acid and for this and other reasons; it is less frequently used than cacao butter. Glycerin suppositories containing ichthammol become insoluble on storage.

Procedure:

1. Calculate for 6 suppositories, that is the amount of Gelatin = $6 \times 14 = 84$ g.
2. Weigh the required amount of gelatin and soak it in enough water until thoroughly softened.

3. Put the soaked gelatin on the calculated amount of glycerin previously weighed in a tarred crucible and on a water bath until gelatin dissolves and a constant weight is obtained.
4. During evaporation the liquid mass should be only gently stirred, rapid stirring produce air bubbles which may appear in the finished suppositories.
5. Remove any skin formed on the surface before pouring.
6. Pour the mass while still hot, into the holes lubricated with liquid paraffin, do not let the melted mass to overflow.

Result: Glycero-gelatin base suppositories was prepared and submitted.

Experiment 16

AIM: - Prepare and submit 20 gm of effervescent granules

Equipments required: - Mortar and pestle, weighing balance, sieve.

Materials required: - Citric acid, Tartaric acid, Sodium bicarbonate, Sucrose

Formula:

Ingredients	Quantity
Sodium bicarbonate	510 gm
Citric acid	180 gm
Tartaric acid	270 gm
Sucrose	150 gm

Send 20 gm

Theory:

Granulation is a method for, improving the flow ability of powder drugs. It includes the converting the powder of the drug into agglomerates of smaller particles (free flowing coarse powder) to be administered as such or to be tabulated or encapsulated. Granulation also allows the addition of flavoring and coloring agents and produces an easily handled, attractive, palatable product.

Effervescent granules: These effervesce on addition to water, and **usually contain mixture of citric, tartaric acids with bicarbonate soda and** usually some medicaments and occasionally sugar. They are dissolved in water for purposes of administration and taken during effervescence or immediately thereafter.

Procedure:

- 1- All the ingredients are finely powdered and passed through sieve No. 60.
- 2- Magnesium sulfate must be exsiccated by heating in a clean dry porcelain dish on direct flame till completely dry.
- 3- The powders are mixed homogenously, massed with 95% ethanol. The produced dough is passed through a sieve No. 10.
- 4- The resultant granules are dried in hot air oven at 40 °C for 4 hours.

Result: Effervescent Granules is prepared and submitted.

Experiment 17

AIM:- Prepare and submit 20 gm of sulfur ointment.

Equipments required:- Mortar and pestle, weighing balance, water bath .

Materials required:- cetostearyl alcohol, hard paraffin, wool fat and white soft paraffin, ppt. Sulfur

Formula:

Formula for sulfur ointment:

Ingredients	Quantity
Precipitated sulfur	100 gm
Simple ointment	900 gm

Formula for simple ointment:

Ingredients	Quantity
wool fat	50 gm
hard paraffin	50 gm
cetostearyl alcohol	50 gm
white soft paraffin	850 gm

Theory:

- Ointments are semi-solid preparations consisting of a medicament or mixture of medicaments dissolved or dispersed in a suitable base.
- They are used as emollients or protective preparations on the skin.
- They are water-immiscible.
- Emulsifiable bases make the ointment miscible with tissue exudates and are more readily removable from the skin by washing.

Procedure :

1. in a porcelain dish, melt cetostearyl alcohol, hard paraffin, wool fat and soft paraffin over a water bath .
2. remove from the heat and stir until cold.
3. powder ppt. Sulfur in a mortar , then incorporate with a portion of the simple ointment until smooth.
4. gradually add the remainder of simple ointment and mix thoroughly.
5. transfer to a clean container and fix a red label.

Uses:

Sulfur is a keratolytic and a mild antiseptic. It is widely employed in the form of lotions or ointments, in the treatment of acne, dandruff, seborrheic conditions and scabies.

Result: Sulfur ointment is prepared and submitted.

Experiment- 18

PREPARATION & SUBMISSION OF FACE POWDER

AIM: -To prepare & submit face powder

REFERENCE: - Hilda Buter, pouches perfumes, cosmetics & soaps. 10th edition. Academic publishers, London. Pg-168

THEORY: -Face powder is use to cover minor imperfection of reduce the shine that appears on the skin due to sebum or perspiration. They are required to give a man, smooth finish to the screen and remain this way for as long as possible. This means that all the ingredients must adhere well to the skin.

Modern products are also required to be long lasting preferably all day & consequently avoid repeated application they should hit rub off onto clothing. To prevent leakage a nylon mesh covers the surface of the powder. The popularity of loose face powders based with the advent of compact (compressed) powders and developments in formation and liquid make up. However, it is still considered by some that loose powder gives a more “professional “finish and there is resurgence in popularity from time to time.

FORMULA-

INGREDIANTS	QUANTITY
Calcium Carbonate	2.5 gm
Magnesium Carbonate	2.5gm
Talc	10 gm
Zinc Stearate	4 gm
Methyl Paraben	0.8gm
Fragrance	0.2gm
Net Weight	20 gm

PROCEDURE: -

1. Dispense the Talc, Zinc Stearate, Calcium Carbonate, Magnesium Carbonate, Frangence, Methyl Paraben and mix for 10-20 mins.
2. Pass the mixture through the sieve (if necessary).
3. Again the mixture is mixed for 10-15 mins.

CONCLUSION: - Hence after performing the experiment we can conclude that face powder was prepared.

Experiment- 19

PREPARATION AND SUBMISSION OF BODY POWDER OR DUSTING POWDER

AIM:-To prepare and submit the body powder or dusting powder.

REFERENCE:- "Cosmetics formulation manufacturing and quality control", p.p prasad. page no-541

THEORY:-Body powder or dusting powder are given or applied on the body are given or applied on the body to reduce friction with cloths that the person would be avoiding infection and rashes. It is composed of talc which is an absorbent and it reduces friction. CaCO_3 act as a protectant. zinc act as a binding agent and boric acid is used as an antiseptic and as additives for color and perfume. Body powder does not required applicator. It is free flowing sieving is to be done.

PROCEDURE:-

1. All the required ingredients were taken and weighed.
2. Then mixed weighed amount of talc, zinc stearate CaCO_3 . At last boric acid and perfume were added in the mortar pestle and triturated it properly.
3. The mixture was triturated and the angle of repose was found as the funnel was placed at a height of 2.5cm.
4. Finally sieving was done.
5. Thus we get body powder.

CONCLUSION:-After performing the experiment we can conclude that the body powder was prepared.

Experiment- 20

PREPERATION AND SUBMISSION OF TOOTH POWDER

AIM: To prepare and evaluate both powder.

Reference: "A Hand book of cosmetics.", B.M. Mithal, R.N. Saha, Vallabh Prakashan, Pg No: 211-215.

Theory: Tooth powder are structurally, the oldest and simplest preparation and they are also the cheapest. The main problem encountered with tooth powders are floating of powders in air during manufacturing formation of cake on storage and uneven distribution in mouth. The oldest tooth powder is reported to be compressed chalk.

Composition: An abrasive e.g.: calcium carbonate, bicalcium phosphate. A surfactant or detergent: e.g.: Sodium lauryl sulphate (SLS). A sweetening agent, e.g.: Sodium saccharine (0.05-0.3%). Flavor. Eg: Spearmint, Peppermint. Color if required. Tooth powders are prepared by just mixing. Ingredients of small quality are premixed and then mixed with often ingredients in ribbon type\agitator type of mixer.

CALCULATION:-

For 10gm of tooth paste, Di-calcium required=4.8gm

For 10gm of tooth paste, Sodium lecocly sulphate required=0.15gm

For 10gm of tooth paste, caboxymethyl cellouose required=0.11gm

For 10gm of tooth paste, Soudium fluride required=0.07gm

For 10gm of tooth paste, Glycerin required=2.2gm

For 10gm of tooth paste, Soudim saccharide required=2.2gm

For 10gm of tooth paste, Flavour required=0.08gm; Ph of toothpaste=8

Foaming stability of tooth paste-----4min 05sec

WORKING FORMULA:

SL NO	Ingredients	Quality Given (For 10 gm)	Quantity Taken (For 50 gm)
1.	Dicalcium Phosphate	4.8 gm	2.4 gm
2.	Sodium Lauryl Sulphate	1.5 gm	0.75 gm
3.	Carboxy Methyl Cellulose	1.1 gm	0.55 gm
4.	Na-Fluride	0.7 gm	0.35 gm
5.	Na-Saccharine	0.2 gm	0.1 gm
6.	Flavor(Clove)	0.8 gm	0.4 gm

Procedure: 4.8 gm of dicalcium phosphate (DCP) 0.15 gm of sodium lauryl sulphate (SLS) was taken in mortar pastel. After that 0.11 gm of CMC was added 0.7 gm of sodium fluoride was also added, the powder mixture was triturated thoroughly. After certain duration of time 0.02 gm of sodium saccharine was added, the trituration was continued until the mixture was obtained in a fine salt. It was sieved and the finest powder was the obtained.

Conclusion: After performing the experiment we can conclude that, we have prepared and evaluated tooth powder.

EXPERIMENT.- 21

PREPARE & SUBMIT OF TOOTH PASTE

AIM: To Prepare & Submit Tooth Paste.

THEORY: Food debris and plaque seen to be the cause for tooth problem. There are tooth surface stains that can be removed in a part if dentifrices. In a part if not all together by the use of a cleaning. Thus, dentifrices are not only cosmetics but are also preservative major for tooth problem.

Minimum Requirement Denitrification is listed below:

1. When used with a tooth brush it should add quality clean teeth of food Debris, plaque & stains.
2. It should have a sensation of clean & freshness in mouth.
3. It should be non-toxic & convenient to use.
4. It should be economic in cost so as to increase its use by all class of society.

Working Formula:

Sl No:	Ingredients	Quantity Given	Uses
1	Dicalcium Phosphate	48 gm	Adhesive
2	Sodium Lauryl Sulphate	1.5 gm	Foaming agent
3	Carboxy Methyl Cellulose	1.1 gm	Gel forming agent
4	Sodium Chloride	0.7gm	Gel vehicle
5	Glycerin	22ml	Sweetening agent
6	Sodium Saccharine	0.2 gm	Chelating agent
7	Flavor (clove)	0.8gm	Flavoring agent

PROCEDURE:

1. 48gm of dicalcium phosphate, 1.5gm of sodium lauryl sulphate were taken in a mortar pestle, to it, 1.1gm Carboxy Methyl Cellulose is added.
2. 22gm of glycerin was added to the powder mixture it was then triturated thoroughly.
3. After 15min, triturate was continued along with addition of 0.2gm of sodium saccharine & 0.7gm of Sodium Chloride.
4. During trituration certain amount of water was added so as to maintain & check its consistency.
5. Appearance the experiment we can conclude that we have prepared & evaluated.

CONCLUSION: So, after the experiment, Tooth-Paste is prepared and submitted.

Experiment- 22

PREPARATION & SUBMISSION OF HAIR GEL

AIM: To prepare and submit hair gel .

REFERENCE: Mittal B.M, Saha R.N, a handbook of cosmetics by Vallabh prakashan, New Delhi pg- 108

THEORY: Gel are transparent and translucent semisolid or solid preparation cosmetics of solution of one or more active ingredients in suitable hydrophobic of and they are high degree of cross linking have relative high yield .Gel are often non grassy and are general applied externally. Aluminium hydroxide gel as well as been used as delivery vehicle for local anesthetics spermatic and dermatological agent. Gel is also used for lubricant of globes and instruments as film former in packing and for conducting of the ferminal of electrocardiograph leads. Gel are also used for lubricant for catches of inserted in to the internal organs are required to be sterile as vehicle for the the persecution of water soluble medicament gel are ideal because ,of their high water content . Product tends to be smooth elegant and procedure. Cooling effect become of evaporation of water. A solid gel produces by cooling a hot solution of gelatin into which medicament has been incorporated as zinc gelatin 1968 for application it is metted and applied with a brush.

Carbomar- Carboxy vinyl polymer of high molecular weight extensive cross linked with poly alkyl sucrose and aqueous dispersion is acidic and forms a gel on neutralization with suitable base .Viscosity is low at ph (3) above 12 relatively low concentration of carbomar is normally sufficient to procedure for neutralized during preparation precaution are necessary of prevent entrapment of air bubble which can aid oxidation in presence of light.

“Hair styling gel “Hair gel is used to important wet look to add ends of strands to long hair together and keep some strands in place.

Procedure:

- 1: 100 ml of water was taken in a beaker and 500 mg of carbomar was weighed.
- 2: The beaker containing water was put on a magnetic stirrer and after addition of almost 200-300 mg of carbomar .Triethylamine was not added any more.
- 3: Mixture was kept for hydration for at least 12 hrs.

Conclusion: After performing the experiment we can conclude that we have prepared and evaluation hair gel.

Experiment- 23

PREPARATION AND SUBMISSION OF SHAVING CREAM

AIM:- To prepare and submit shaving cream.

REFERENCE:-Mittal B.H, Saha R.N. A handbook of cosmetics vallabh prakashan, New Delhi, Pg-172.

THEORY:- Shaving cream preparations are used for softening the bearded for wet shaving and also to produce rich foams. Brushless shaving creams are preparation in which leather with brush is omitted. After washing off the face with soap and warm water, these shaving creams are applied to keep the bearded soft, till the shaving is completed. Initial washing helps in defaulting and makes the hair soften. The creams function is to prevent the keratin from drying and miscible with water for even spreading. They mainly consists of stearate, soaps and additionally contain oil, humectant viscosity enhancing agent. The fatty sub stains should be atleast 20% incorporating of some waxes can enhance the viscosity and it is required, as consistency is important for proper application. It should also contain perfume and preservatives.

PROCEDURE:-

1. 10 ml of paraffin oil, 1.5 gm of stearic acid, 1gm of Cetyl alcohol was taken in beaker.
2. It was heated on water bath at 80-85°C with constant stirring.
3. 2ml of triethanolamine, 1gm of sodium lauryl sulfate and 45.4 gm of water was taken in a beaker. It was heated on a water-bath at 60-65°C with constant stirring.
4. 5ml of propylene glycol, 0.2 mg of propyl paraben was taken in a beaker. This mixture was added with triethanolamine, sodium lauryl sulfate and water.
5. 0.6gm of carbomer and 20ml of water was taken in a beaker. It was heated on a water-bath or 100°C with vigorously shaking.
6. The mixture of triethanolamine, sodium lauryl sulphate and water was added to paraffin oil, stearic acid and Cetyl alcohol with constant stirring at 65°C.
7. The mixture of paraffin oil, stearic acid, Cetyl alcohol was added and mixed with mixtures of carbomer and water containing propylene glycol and Propyl paraben. To it, few drops of perfume were added.
8. Constant stirring was done, until the desired product of shaving cream was achieved.

CONCLUSION:- After performing the experiment, we can conclude that the prepared and evaluated shaving cream.

IV SEMESTER (II-B.PHARM)

PHYSICAL PHARMACEUTICS - II

PRACTICAL LAB MANUAL

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PHYSICAL PHARMACEUTICS-II

(PRACTICAL MANUAL) SECOND YEAR (IV- SEMESTER)

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1. DETERMINE THE ANGLE OF REPOSE AND INFLUENCE OF LUBRICANT ON ANGLE OF REPOSE

AIM

To determine the effect of glidants on lubricants of angle of repose

REQUIREMENTS

- Lactose powder
- Starch paste
- Talc
- Mortar and pestle
- Funnel, stand

PRINCIPLE

Lubricants are glidants of friction during tablet, ejection between the starch of the tablet and the walls of the die cavity. The most widely used lubricants have been steric acid and steric acid derivatives such as calcium and magnesium stearate and talc. Glidants are intended to produce flow of the tablet granulation of powder materials by reducing friction between the particles. The most widely used glidants have been derivation of talc and corn starch.

PROCEDURE

- ❖ Select a glass funnel which has a round shape of 15-30mm of diameter with flat edge
 - ❖ Fix the funnel with a clamp (on the ring)
 - ❖ Place the glass plate on the ring and arrange it below the glass funnel
 - ❖ Keep on graph paper on the glass funnel
 - ❖ Weigh approximately 100gm of granules
 - ❖ Pour the granules while blocking the orifice of the funnel be thumb
 - ❖ Remove the thumb the granules load at flow down into the graph paper and form a cone shaped
-

- ❖ Adjust the thumb the funnel clamp so that the gap between the bottom of the funnel peak of the powder pile is about 3mm
- ❖ Repeat the 5-7 steps and approximate graph is maintained
- ❖ Finally pour the granules back into funnel and allow to flow
- ❖ Mark four points which are opposite to each other on the circular base on the graph paper
- ❖ Record the readings in table, this value is the diameter calculate the radius in cm
- ❖ Measure the height of the pile using two rulers
- ❖ Keep one ruler vertically and another horizontally to touch the peak of the pile, then read the value for the vertical scale.
- ❖ Substitutes the value in equation to obtained the angle of repose, generally the (h/r) measure is the angle of repose data were plotted semi-log paper and copies of curves made available for the purpose of calculating angle
- ❖ Repeat the procedure 2 more time and take on average

REPORT

The angle of repose of the given granules (without glidant) =

The concentration is

The effect of glidants of lubricants of angle of repose is =

Inference is that the flow of granules

TRIAL	HEIGHT (cm)	RADIUS (r) (cm)	h/r	Angle of Repose $\theta = \tan^{-1} h/r$
I				
II				
Average angle of Repose =				

2. DETERMINATION OF BULK DENSITY, TRUE DENSITY AND PERCENTAGE POROSITY

AIM

To determine the bulk density, true density and percentage porosity of the given granules

PRINCIPLE

- It is defined mathematically as

Bulk density = /

- When particles are packed loosely lots of gaps between the particles are observed. Hence the bulk volume increases by making the powder light based on bulk volume powder are classified as light and heavy
- light powder have high bulk volumes on the other hand smaller particles the powder assume low bulk volume or high bulk volume density such powder are called heavy powder. The bulk density depends on particle size distribution, shape, and cohesiveness of particles.
- True density is the density of the powder itself

True density = /

- The density depend on the type of atom in a molecular rearrangement of atoms in a molecule and arrangement of molecule in the sample volume occupied by voids and the intra particle pores are not included in the most common method used in the determination of true density or gas displacement or liquid displacement method.
 - This method is used to select a solvent in which the powder is insoluble
-

PROCEDURE:

- ✓ Approximately 20gm of powder is transferred to a 500ml cylinder and tap mechanically or by tapping device until a constant volume is obtained thus volume is bulk volume and the void space among powder particle

True density:

Determination of true density of the material by solvent displacement method

- Weigh accurately a clean and dry density bottle
- Take the weight of density bottle with small quantity of powder sample
- Now fill the density bottle by solvent without removing the powder material
- Calculate the true density of given powder sample

Determination of percentage porosity

- Porosity is defined as the void volume to the bulk volume of the granules

$$\text{Porosity} = 1 - \frac{\text{Bulk Volume}}{\text{True Volume}}$$

REPORT

- ✓ The bulk density of the given sample of granules was found to be = g/cm^3
 - ✓ The true density of a given powder was found to be = g/cm^3
 - ✓ The percentage porosity of the a given powder is = %
-

3. DETERMINATION OF VISCOSITY OF LIQUID USING OSTWALD'S VISCOMETER

AIM:

To determine the viscosity of the unknown liquid by using Ostwald's viscometer

REQUIREMENTS:

- Ostwald's viscometer
- Stop clock
- Specific gravity bottle
- Sample
- Distilled water

PRINCIPLE:

The force of friction with one part of a liquid offers to another part of the liquid is called viscosity. For measuring the viscosity coefficient Ostwald's viscometer method is used which is based on poiseuilles's law. According to this law, the rate of flow of liquid through a capillary tube having viscosity coefficient (η)

$$\eta = \frac{\pi r^4 t \Delta P}{8LV}$$

Where,

V= volume of liquid (ml)

t= flow of time in seconds through capillary (in second)

R= radius of capillary (cm)

η = viscosity coefficient (poise)

P= hydrostatic pressure

L= length

- Since the hydrostatic pressure (driving force) of the liquid is given by

$$\eta = dgh$$

Where,

h= height of the column

d= density of the liquid

PROCEDURE:

- Wash the relative density bottle with distilled water and dried.
- Take the weight of empty bottle and filled given liquid
- Clean and rinse the viscometer properly with distilled water
- Fix the viscometer vertically in the stand and filled the specific amount of given unknown liquid in viscometer
- Time of flow recorded when the liquid starts to flow from the mark c and d above and below the bulb a. the experiment repeated 3-4 times to get viscosity of the given unknown liquid.

LIQUID	FLOW TIME IN (SEC)			AVERAGE (SEC)	DENSITY (g/ml)
	1	2	3		
distilled water					
given sample					

REPORT:

The viscosity of the unknown liquid is = centipoise

4. DETERMINATION OF SEDIMENTATION VOLUME WITH EFFECT OF DIFFERENT SUSPENDING AGENT

AIM:

To determine the sedimentation volume with effect of different suspending agent.

PRINCIPLE:

SUSPENSION

Pharmaceutical suspension may be defined as a coarse dispersion in which insoluble solids are suspended in liquid medium. It is also known as heterogeneous system (or) more precisely biphasic system. The insoluble solids may have size range from 10-10000 μ m and liquid medium is normally water or a water based vehicle.

SUSPENDING AGENT

Suspending agent is defined as physiologically inert substance which increases the viscosity when added to suspensions. It helps in the keeping the dispersed particles. Suspended thus there enhanced the physical stability and re-dispersion of the sediment or shaking.

PHYSICAL STABILITY

Physical stability may be defined as a condition in which particles remain uniformly distributed throughout the dispersion with any signs of sedimentation. In practice Physical stability may be defined as a condition in which particles should be easily re-suspended by a moderate shaking. If they settle suspensions when kept aside. The solids tend to settle at the bottom of the container due to gravitational pull on the particles of higher size. It is not possible to prevent the sedimentation volume and its case of re-dispersion are the common evaluation procedure for assessing the physical stability. The two sedimentation parameters are employed such as

1. Sedimentation volume
 2. Degree of flocculation
-

SEDIMENTATION VOLUME:

- Sedimentation volume is defined as

$$F = \frac{\text{Sedimentation volume}}{\text{Height of cylinder}}$$

- When a suspension is taken in a measuring cylinder volume and height is proportional and height can be conveniently measured through the term volume is included in the terminology sedimentation volume “F” is a dimension less number. Most pharmaceutical suspension has an “F” value less than one. If $f=1$ the product has no sediment and no clear supernatant on standing which is an ideal condition. Normally “F” value lies between 0 and 1. Sometimes the network of flow is loose and fluffy and ultimate volume of sediment increase. In this situation “F” value will be greater than one.

PROCEDURE:

1. Weigh 5gm of calcium carbonate and place in a mortar and add small quantity of water and triturate the sample. After suspending the powder uniformly transfer the suspension into a 100ml measuring cylinder make up the volume to 100ml with distilled water.
2. Separately prepare 5% w/v of calcium carbonate suspension with 1% of different suspending agent such as bentonite, methyl cellulose, respectively in different vessel add small quantity of water and triturate well. After powder is uniformly suspended transfer the suspension into separate 100ml measuring cylinder.
3. Make up the volume to 100ml with distilled water
4. Shake the suspension simultaneously and kept aside
5. Note the volume of sediment at time periods 0,10,20,30,...60 minutes. Calculate the sedimentation volume
6. Draw the plot by taking “F” values on “Y” axis and the time on “X” axis.

REPORT:

5% calcium carbonate suspension 1% carboxy methyl cellulose as suspending was found to be more physically stable compared with other suspending agent.

5. DETERMINATION OF SEDIMENTATION VOLUME WITH EFFECT OF DIFFERENT CONCENTRATION OF SINGLE SUSPENDING AGENT

AIM:

Determination of Sedimentation volume With Effect of Different Concentration of Single Suspending Agent

PRINCIPLE:

SUSPENSION

Pharmaceutical suspension may be defined as a coarse dispersion in which insoluble solids are suspended in liquid medium. It is also known as heterogeneous system (or) more precisely biphasic system. The insoluble solids may have size range from 10-10000 μ m and liquid medium is normally water or a water based vehicle.

SUSPENDING AGENT

Suspending agent is defined as physiologically inert substance which increases the viscosity when added to suspensions. It helps in the keeping the dispersed particles. Suspended thus there enhanced the physical stability and re-dispersion of the sediment or shaking.

PHYSICAL STABILITY

Physical stability may be defined as a condition in which particles remain uniformly distributed throughout the dispersion with any signs of sedimentation.

In practice Physical stability may be defined as a condition in which particles should be easily re-suspended by a moderate shaking. If they settle suspensions when kept aside. The solids tend to settle at the bottom of the container due to gravitational pull on the particles of higher size. It is not possible to prevent the sedimentation volume and its case of re-dispersion is the common evaluation procedure for assessing the physical stability. The two sedimentation parameters are employed such as

1. Sedimentation volume

2. Degree of flocculation

SEDIMENTATION VOLUME:

- Sedimentation volume is defined as

$$F = \frac{\text{Sedimentation volume}}{\text{Total volume}}$$

- When a suspension is taken in a measuring cylinder volume and height is proportional and height can be conveniently measured through the term volume is included in the terminology sedimentation volume “F” is a dimension less number. Most pharmaceutical suspension has an “F” value less than one. If $f=1$ the product has no sediment and no clear supernatant on standing which is an ideal condition. Normally “F” value lies between 0 and 1. Sometimes the network of flow is loose and fluffy and ultimate volume of sediment increase. In this situation “F” value will be greater than one.

PROCEDURE:

1. Separately prepare 5% w/v of calcium carbonate suspension with 0.5%, 1%, 1.5%, 2 % of single suspending agent such as (bentonite) in different vessel. Add small quantity of water and triturate well. After powder is uniformly suspended transfer the suspension into separate 100ml measuring cylinder.
2. Makeup the volume to 100ml with distilled water
3. Shake the suspension simultaneously and kept aside
4. Note the volume of sediment at time periods 0, 10, 20, 30,...60 minutes. Calculate the sedimentation volume
5. Draw the plot by taking “F” values on “Y” axis and the time on “X” axis.

REPORT:

- Increase the concentration of suspending agent and also increase the viscosity so lower the sedimentation volume
 - The concentration of single suspending agent bentonite has more physical stable.
-

Sedimentation volume of 5% calcium carbonate suspension with 5% bentonite

S.NO	TIME (MIN)	$F = V_U/V_0$
1		
2		
3		
4		
5		
6		
7		



6. DETERMINATION OF PARTICLE SIZE DISTRIBUTION BY SIEVING METHOD

AIM:

To determine the average particle size and find out their distribution pattern for the given granules by sieve analysis method.

PRINCIPLE:

Sieve method gives sieve diameter, sieve diameter is defined as the diameter of the sphere that possess through the sieve aperture as the asymmetric particle sieve method directly give weight distribution. Particles having size range from 50 and 1500 μm are estimated by sieving method. In this method, the size is expressed as d_{sieve} . The sieving method finds application in dosage and development of tablets and capsules. Normally 15 percent of fine powder (passed through mesh 100) should be present in granulated material to get a proper flow of material and achieve good compaction in tableting. Therefore, percent of coarse and fine can be quickly estimated. Sieves for pharmaceutical testing are constructed from wire cloth with square meshes, woven from wire of brass, bronze, stainless steel or any other suitable material.

Designations and Dimensions of I.P specification sieves

Sieve Number	Aperture Size Micrometer	Sieve Number	Aperture Size Micrometer
10	1700	44	325
12	1400	60	250
16	1000	85	35
22	710	100	36
25	600	120	34
30	500	150	36
36	425	170	35



Advantages of sieving method

1. It is inexpensive, simple and rapid with reproducible results.
2. Sieving method is useful when particles are having size range between 50 and 1500 μm .

Disadvantage of sieving method

1. Lower limit of the particle size is 50 μm .
2. If the powder is not dry, apertures become clogged with particles leading to improper sieving.
3. During shaking, attrition occurs causing size reduction of particles. This leads to errors in estimation.

Factors influencing the sieving method

Factors influencing sieving are weight of sample, duration of shaking and type of motion. The types of motion influencing sieving are vibratory motion, (most efficient), side tap motion, bottom pat motion, rotary motion with tap and rotary motion. The type of motion standardized. Care should be taken in order to get reproducible results.

PROCEDURE:

1. Standard sieves set is selected (sieve no: 10, 22, 36, 44, 65, 80, 100,120) arrange them in such manner that the coarsest remains at the top and finest at the bottom.
2. Weigh approximately 50g of sample place the sample on the coarsest sieve no.10.
3. Fix the above sieves set on hand sieve shaker and shaken for 20 minutes.
4. Collect the Sample retained on each sieve into a paper, weigh all the sample.
5. Report the weights retained on each sieve in the table against corresponding sieve number.

REPORT:

The average diameter of the given granules was found to be 493.47 μm .

7. CALIBRATION OF EYE PIECE MICROMETER

STANDARD STAGE MICROMETER:

- ✓ Standard stage micrometre is used to calibration of eye piece micrometre. Eye piece micrometre is a glass slide (7.5cm into 2.5cm) which has the scales engraved in the scale usefully 0.1mm is length. 1mm divided into 100 divisions. Thus smallest division least count of the stage micrometre represents 0.01mm or 10 μ m length.
- ✓ In this experiment in the optical combination of 10x eye piece and 45x objective is used
- ✓ The stage micrometre is least on the stage of the microscope. The objective is position to the centre of objective
- ✓ Initially disc focus low power the scale of stage micro meter observed (100 divisions)
- ✓ Now the objective is focus to high power (45x)
- ✓ Two points were selected one point on the left side where divisions both scales coincide and another point on the right side
- ✓ The number of small division that is eye piece were counted and big division stage micrometre were counted and recorded

$$1 \text{ eye piece} = y/x \times 0.01\text{mm}$$

$$1 \text{ eye piece} = y/x \times 10 \mu\text{m}$$

PROCEDURE:

Counting of the sample

1. A small portion of given sample transfer to a clean slide
 2. One (or) two drops of liquid paraffin is added to the slide
 3. The sample is dispersed uniformly with help of brush and particles should be in depended and distribution should be uniform
 4. The cover slip is placed carefully entrancement of air bubbles is avoided
 5. The slide is placed the stage of microscope
-

Measurement of particle size:

- The slide is focus in low power (10x) the presence of individual particle is absorb (if aggregation or lumps are present the sample should be mounted again)
- The size of the each particles measure is terms of eye piece division
- A total 300 particles should be considered for size distribution analysis. Ideally 625 particles measure according to BPC.

REPORT:

From the graph it was found the particles were distributed uniformly from size range of 0-400 μ m.

Size Range (μm)	Mean Size (D) μm	Number Of Globules In Each Size Range	% Number Of Globules	Cumulative % Number Of Globules	Number Size
0-50					
50-100					
100-150					
150-200					
200-250					

8. DETERMINATION OF REACTION RATE CONSTANT FIRST ORDER

AIM:

To determine the reactant rate constant and half-life and the ester (methyl acetate or number of globules) at 0.5M HCL at room temperature.

APPARATUS AND CHEMICALS REQUIRED:

- Conical flask (250 ml)
- 10 ml volumetric pipette
- Burette
- Ethyl acetate or methyl acetate
- Test tube
- 0.5 N HCL solution
- 0.25 N NAOH solution
- Phenolphthalein indicator
- Ice cold water

PROCEDURE:

Preparation of hydrochloric acid solution (0.5N) IP:

Solutions of any normality XN may be prepared by diluting 85 x ml of HCL to 1000ml with water. Measure 850 ml of distilled water into a 1000ml volumetric flask. Add 42.5 ml of conc.HCL and slowly added. Finally make up the water in 1000ml.

Preparation of NAOH solution (0.25 N) IP:

Solutions of any normally XN may be prepared by dissolving 40 x gm of NAOH in water and diluting to 1000ml. weigh 10gm of NAOH and transferd into 1000ml volumetric flask. Add water slowly with stirring finally makeup the water into 1000ml.

PROCEDURE:

KINETIC METHOD

- ❖ 100ml of 0.5N HCL solution is measured and transferred into a 250ml conical flask
 - ❖ It should be kept in the water bath for equilibrium (do not heat)
 - ❖ 10ml of the given ester is transferred into the test tube and kept in the water bath for equilibrium. Normally it takes 10 minutes
 - ❖ The acid solution is mixed with ester sample thoroughly and kept in water bath
 - ❖ Immediately after mixing 5ml of the mixture is withdrawn using the pipette and transferred into a conical flask containing 10ml ice water (0 time)
 - ❖ A few drops of phenolphthalein indicator are added to the mixture
 - ❖ The reaction mixture is titrated against 0.25N NaOH solution. This volume of alkali consumed represents V_0 .
 - ❖ 5ml samples are withdrawn periodically at 10, 20, 30, 40, 50, 60, 75 minutes. The volume consumed at each time interval represents V_t
 - ❖ The reaction mixture is heated in a water bath at 60°C for 20 minutes
 - ❖ The mixture is cooled to room temperature
 - ❖ 5ml of sample is withdrawn and transferred into the conical flask containing 10 ml ice cold water. The titration is repeated and this volume represents V_{α} .
-

REPORT:

The reaction rate constant (k) of the given data (methyl acetate or ethyl acetate) in 0.5N HCL acid is

From graphical method= minutes

From substitution method= minutes

The half -life ($t_{1/2}$) of the given ester (methyl acetate or ethyl acetate) in 0.5N HCL acid is

From graphical method=

From substitution method=

9. DETERMINATION OF VISCOSITY OF SEMISOILD BY USING BROOKEFIELD VISCOMETER

AIM:

To determine the viscosity of semisolid by using Brooke field viscometer

PRINCIPLE:

Newton was the first to study the flow properties of liquids in quantitative terms liquids that obey newton's law of flow are called as Newtonian fluids

$$F = \eta G$$

Shear stress- shear rate

Relationship is normally in the form of a curve rheogram or consistency curve. When data are plotted by taking "F" on x-axis and "G" on y-axis, a flow curve is obtained. The rheogram passes through the origin and the slope given the coefficient of viscosity system that follow this linear relationship are called as Newtonian fluids. This class includes liquids such as water, glycerine, chloroform, solutions of syrups, very dilute colloidal solution. Simple liquids exhibit Newtonian flow. Rheological properties of heterogeneous dispersions such as emulsions, suspensions and semisolid are more complex and do not obey newton's equation of flow based on the pattern of consistency curve, Non-Newtonian fluids are categorized as

- ❖ Plastic flow
- ❖ Pseudo plastic flow
- ❖ Dilatant flow

PROCEDURE:

- Prepare bentonite magma (5% w/v), methyl cellulose (2% w/v) and mineral oil. They show Non-Newtonian rheological profile
 - Measure the viscosity of these liquids using a Brookfield viscometer and observe the thixotrophy phenomenon
-

-
- Place the spindle with the correct number listed in the data sheet in each liquid and rotate the spindle at the speeds indicated. Once the dial reading has stabilized, record the values of viscosity in (cps).

CALCULATION:

Viscosity in cps= dial reading x factor

S. No	Spindle Speed	Factors	Dial Reading	Viscosity (F × Dr)
1.	6	1000		
2.	12	500		
3.	30	200		
4.	60	100		
5.	30	200		
6.	12	500		
7.	6	1000		

REPORT:

The viscosity of the given sample was found to be= centipoise (cps)

PROCEDURE:

Preparation of ethyl acetate solution (0.05N)

The molecular weight of ethyl acetate is 88.10 density is 0.90g/ml. percentage purity is 99%. Measure 50 ml of Ethyl acetate and transfer into 1000ml volumetric flask dilute to 1000ml with distilled water.

Preparation of hydrochloric acid solution (0.02N)

Solutions of any normality XN may be prepared by diluting 85 ml of hydrochloric acid to 1000ml with water. Measure 850 ml of distilled water into 1000ml volumetric flask. Add 1.7ml of concentrated hydrochloric acid slowly and shake. Finally make up the volume to the mark.

Preparation of sodium hydroxide solution (0.05N)

Weigh 2.0gm of sodium hydroxide in water and transfer into 1000ml volumetric flask. Add water slowly with continuous stirring, while cooling the flask under running tap water. Add sufficient water to make 1000ml. allow it to stand overnight and pour off the clear liquid into a bottle. This clear solution is used.

Kinetic method:

- ✓ Measure 50ml of 0.05N sodium hydroxide solution and transfer into a conical flask. Keep it in a water bath for equilibrium at room temperature.
 - ✓ Measure 50ml of 0.05N of the given ester and transfer into a conical flask. Keep it in above water bath for equilibrium. Normally it takes about 10 minutes.
 - ✓ Mix the alkali and ester solution thoroughly and keep in same water bath.
 - ✓ Immediately after mixing, withdrawn a 10ml sample of the mixture with pipette and transfer into a conical flask containing 10ml ice cold water
 - ✓ Add few drops of Phenolphthalein indicator
 - ✓ Titrate against 0.02N hydrochloric acid. This titer value times $t=0$ corresponds to the original concentration "a" report the results
-

- ✓ Periodically withdrawn samples at 5, 10, 15, 20, 25, 30 minutes time periods. Repeat the steps 4 to 6. These titer values denote the amount of sodium hydroxide or ethyl acetate remain unreacted ie, (a-x) at time. Record the results
- ✓ Substitute the values in integral equation and calculate the reaction rate constant (K_2). These values will more or less constant
- ✓ Calculate the average of the reaction rate constant (K_2)
- ✓ Draw a plot by taking x/a (a-x) on y-axis and time on x-axis
- ✓ Estimate the slope. This slope same as K_2 value.

Calculation:

time (min)	volume of HCL consumed		volume of HCL (ml)	concentration in mol/liter a or (a-x)	x= a(a-x)	x/a(a-x)	$K_2 = x/at$ (a-x) liter/mol
	initial (ml)	final (ml)					
00							
5							
10							
15							
20							
25							
30							

REPORT:

The reaction rate constant (K_2) of the given ester (methyl acetate or ethyl acetate) in 0.025N NAOH at room temperature

From graphical method= minutes

From substitution method= minutes

The half -life ($t_{1/2}$) of the given ester (methyl acetate or ethyl acetate) in 0.025N NAOH at room temperature

From graphical method=

From substitution method=

11. ACCELERATED STABILITY STUDIES

AIM:

To determine the shelf-life of the product. If stored at 25°C from the given data.

- A pharmaceutical product needs to be physically, chemically, therapeutically, toxicologically and microbiologically stable throughout its shelf-life. The pharmaceutical companies do stability testing for estimating the shelf-life and based on this the expiry date is given for the product.
- The real time studies at recommended condition are ideal method for predicting shelf-life often the studies are designed to increase the rate of chemical degradation or physical change of pharmaceutical products by using exaggerated storage conditions. This is known as accelerated stability testing. The pharmaceutical products are subjected to higher temperature and humidity conditions for accelerating the degradation. However the results of accelerated testing are not always predictive of physical changes and potency.
- The pharmacopoeia specifies certain storage conditions. The following table gives the details as specified in Indian pharmacopoeia.

Storage Condition	Meaning
Cold	any temperature not exceeding 8°C (2-8°C)
Cool	any temperature between 8-25°C
Warm	any temperature between 30-40°C
Excessive Heat	any temperature above 40°C

PRINCIPLE:

Though the medicinal products need to be physically, chemically, therapeutically, toxicologically and microbiologically stable. The chemical instability is most often the main consideration for determining the shelf-life or expiry date. The medicinal products are stored at higher temperature conditions to accelerate the degradation rate. This is known as accelerated stability testing. The rate of chemical reaction increases by 2-3 folds for every rise in 10°C at room temperature. The Arrhenius equation plot (log k vs 1/t) from the equation

$$\text{Log}(k) = \text{log}(A) - [K/2.303 RT]$$

Where,

K= rate constant

R= gas constant

T= absolute temperature

E= energy of activation is used to find out the reaction rate constant at 25⁰C.

PROCEDURE:

- The order of drug decomposition reaction is determined first by plotting curve. Percent potency retained VS time. Here it is first order
- The k value is determined for each temperature curve.
- The Arrhenius plot is drawn log k vs 1/t
- The value at desired temperature is determined by extrapolating Arrhenius equation
- The value of k is placed in the first order rate equation and is calculated.

Three drug products were kept at 4⁰C ± 2⁰C/ 75% RH ± 5% RH

Storage Period In Months	Potency Retained Product-I	Potency Retained Product-II	Potency Retained Product-III
0			
3			
6			

REPORT:

The shelf-life of the medicinal product is

Product-I $K=$

Product-II $K=$

Product -III $K=$

The best product is=

PHARMACEUTICAL ENGINEERING
Lab Manual

B. Pharmacy II Year III Semester

PT 397 - PHARMACEUTICAL ENGINEERING (Practical) 4 Hours/week

- I. Determination of radiation constant of brass, iron, unpainted and painted glass.
- II. Steam distillation – To calculate the efficiency of steam distillation.
- III. To determine the overall heat transfer coefficient by heat exchanger.
- IV. Construction of drying curves (for calcium carbonate and starch).
- V. Determination of moisture content and loss on drying.
- VI. Determination of humidity of air – i) From wet and dry bulb temperatures –use of Dew point method.
- VII. Description of Construction working and application of Pharmaceutical Machinery such as rotary tablet machine, fluidized bed coater, fluid energy mill, de humidifier.
- VIII. Size analysis by sieving – To evaluate size distribution of tablet granulations –
+Construction of various size frequency curves including arithmetic and logarithmic probability plots.
- IX. Size reduction: To verify the laws of size reduction using ball mill and determining Kicks, Rittinger's, Bond's coefficients, power requirement and critical speed of Ball Mill.
- X. Demonstration of colloid mill, planetary mixer, fluidized bed dryer, freeze dryer and such othermajor equipment.
- XI. Factors affecting Rate of Filtration and Evaporation (Surface area, Concentration and Thickness/ viscosity
- XII. To study the effect of time on the Rate of Crystallization.
- XIII. To calculate the uniformity Index for given sample by using Double Cone Blender.

1. DETERMINATION OF RADIATION CONSTANT OF BRASS

AIM: -To determine the radiation constant of Brass cylinder.

REQUIREMENT:-

Brass Cylinder with hole or cavity.

Thermometer (360°C)

Burner

Weighing Balance

Stop watch

Screw gauge

Graph Paper

PRINCIPLE: - Heat transfer is a major unit operation of pharmacy. Heat flows from a region of higher temperature to a region of low temperature. Heat may flow by one or more of the three basic mechanisms.

- a) Conduction is a process in which heat flow in a body is achieved by the transfer of the momentum of the individual atoms or molecules without mixing.
- b) Convection is a process in which heat flow is achieved by actual mixing of warmer portions with cooler portions of the same materials.
- c) Radiation is a process in which heat flows through spaces by means of electromagnetic waves. It is also called as thermal radiation.

In this system, the heat loss through convection is neglected, since movement of particles is negligible. As the metal cylinder is freely suspended without any contact with the metal, the heat loss through conduction is considered minimum. Thus heat loss by radiation is highlighted. Stefan-Boltzmann law gives the rate of radiation emitted by a body.

$$q = bAT^4$$

Where, q = Energy radiated per second, W (or J/s)

A = Area of radiating surface, m^2

T = Absolute temperature of the radiating surface, K

B = Constant, $W/m^2.K^4$

The difference in the temperature of hot body and ambient is the temperature gradient for the heat loss by radiation. The radiation constant (α) is calculated using the following equation:

$$Ms \frac{dq}{dt} = \alpha A [(T_1/100)^4 - (T_2/100)^4] + \beta A (T_1 - T_2)^{1.23}$$

Where,

M = Mass of the metal cylinder, w g

s = Specific heat of the metal, J/Kg.K

(dq/dt) = Rate of heat loss by metal cylinder, W/s

T₁ = Temperature of the metal body, K

T₂ = Temperature of the ambient (room temperature), K

α = Radiation constant, W/m².K⁴

β = Convection factor

A = Surface area for heat transfer, m²

PROCEDURE:-

- 1) Take a Brass cylinder whose surface is smooth.
- 2) Measure the weight, area and radius of the cylinder.
- 3) Place the cylinder on a tripod stand and heat it for about 300⁰C
- 4) Now hold the cylinder with the tongs and place on a non conducting surface (wood/glass) without touching any surface.
- 5) Note the temperature reading for every five minutes, using stop watch.
- 6) Plot a graph between time on X-axis and temperature on Y axis.
- 7) Find out the slopes dq/dt at various arbitrary temperature.
- 8) Calculate radiation constant.

OBSERVATIONS AND CALCULATIONS:

Time, mins	Temperature, °C	Time, mins	Temperature, °C	Time, mins	Temperature, °C

Weight of the brass cylinder =

Height of the brass cylinder =

Diameter of the brass cylinder =

Radius of the brass cylinder =

Surface area of the brass cylinder =

REPORT:-

2. DETERMINATION OF RADIATION CONSTANT OF IRON

AIM: -To determine the radiation constant of Iron cylinder.

REQUIREMENT:-

Iron Cylinder with hole or cavity.

Thermometer (360°C)

Burner

Weighing Balance

Stop watch

Screw gauge

Graph Paper

PRINCIPLE: - Heat transfer is a major unit operation of pharmacy. Heat flows from a region of higher temperature to a region of low temperature. Heat may flow by one or more of the three basic mechanisms.

- a) Conduction is a process in which heat flow in a body is achieved by the transfer of the momentum of the individual atoms or molecules without mixing.
- b) Convection is a process in which heat flow is achieved by actual mixing of warmer portions with cooler portions of the same materials.
- c) Radiation is a process in which heat flows through spaces by means of electromagnetic waves. It is also called as thermal radiation.

In this system, the heat loss through convection is neglected, since movement of particles is negligible. As the metal cylinder is freely suspended without any contact with the metal, the heat loss through conduction is considered minimum. Thus heat loss by radiation is highlighted. Stefan-Boltzmann law gives the rate of radiation emitted by a body.

$$q = bAT^4$$

Where, q = Energy radiated per second, W (or J/s)

A = Area of radiating surface, m^2

T = Absolute temperature of the radiating surface, K

B = Constant, $W/m^2.K^4$

The difference in the temperature of hot body and ambient is the temperature gradient for the heat loss by radiation. The radiation constant (α) is calculated using the following equation:

$$Ms \frac{dq}{dt} = \alpha A [(T_1/100)^4 - (T_2/100)^4] + \beta A (T_1 - T_2)^{1.23}$$

Where,

M = Mass of the metal cylinder, w g

s = Specific heat of the metal, J/Kg.K

(dq/dt) = Rate of heat loss by metal cylinder, W/s

T₁ = Temperature of the metal body, K

T₂ = Temperature of the ambient (room temperature), K

α = Radiation constant, W/m².K⁴

β = Convection factor

A = Surface area for heat transfer, m²

PROCEDURE:-

1. Take a Iron cylinder whose surface is smooth.
2. Measure the weight, area and radius of the cylinder.
3. Place the cylinder on a tripod stand and heat it for about 300⁰C
4. Now hold the cylinder with the tongs and place on a non conducting surface (wood/glass) without touching any surface.
5. Note the temperature reading for every five minutes, using stop watch.
6. Plot a graph between temperature on Y axis and time on X-axis.
7. Find out the slopes dq/dt at various arbitrary temperature.
8. Calculate radiation constant.

OBSERVATIONS AND CALCULATIONS:

Time, mins	Temperature, °C	Time, mins	Temperature, °C	Time, mins	Temperature, °C

Weight of the iron cylinder =

Height of the iron cylinder =

Diameter of the iron cylinder =

Radius of the iron cylinder =

Surface area of the iron cylinder =

REPORT:-

3. DETERMINATION OF RADIATION CONSTANT OF COPPER

AIM: -To determine the radiation constant of Copper cylinder.

REQUIREMENT:-

Copper Cylinder with hole or cavity.

Thermometer (360°C)

Burner

Weighing Balance

Stop watch

Screw gauge

Graph Paper

PRINCIPLE: - Heat transfer is a major unit operation of pharmacy. Heat flows from a region of higher temperature to a region of low temperature. Heat may flow by one or more of the three basic mechanisms.

- d) Conduction is a process in which heat flow in a body is achieved by the transfer of the momentum of the individual atoms or molecules without mixing.
- e) Convection is a process in which heat flow is achieved by actual mixing of warmer portions with cooler portions of the same materials.
- f) Radiation is a process in which heat flows through spaces by means of electromagnetic waves. It is also called as thermal radiation.

In this system, the heat loss through convection is neglected, since movement of particles is negligible. As the metal cylinder is freely suspended without any contact with the metal, the heat loss through conduction is considered minimum. Thus heat loss by radiation is highlighted. Stefan-Boltzmann law gives the rate of radiation emitted by a body.

$$q = bAT^4$$

Where, q = Energy radiated per second, W (or J/s)

A = Area of radiating surface, m²

T = Absolute temperature of the radiating surface, K

B = Constant, W/m².K⁴

The difference in the temperature of hot body and ambient is the temperature gradient for the heat loss by radiation. The radiation constant (α) is calculated using the following equation:

$$Ms \frac{dq}{dt} = \alpha A [(T_1/100)^4 - (T_2/100)^4] + \beta A (T_1 - T_2)^{1.23}$$

Where,

M = Mass of the metal cylinder, w g

s = Specific heat of the metal, J/Kg.K

(dq/dt) = Rate of heat loss by metal cylinder, W/s

T₁ = Temperature of the metal body, K

T₂ = Temperature of the ambient (room temperature), K

α = Radiation constant, W/m².K⁴

β = Convection factor

A = Surface area for heat transfer, m²

PROCEDURE:-

- 1) Take a Copper cylinder whose surface is smooth.
- 2) Measure the weight, area and radius of the cylinder.
- 3) Place the cylinder on a tripod stand and heat it for about 300⁰C
- 4) Now hold the cylinder with the tongs and place on a non-conducting surface (wood/glass) without touching any surface.
- 5) Note the temperature reading for every five minutes, using stop watch.
- 6) Plot a graph between time on X-axis and temperature on Y axis.
- 7) Find out the slopes dq/dt at various arbitrary temperature.
- 8) Calculate radiation constant.

OBSERVATIONS AND CALCULATIONS:

Time, mins	Temperature, °C	Time, mins	Temperature, °C	Time, mins	Temperature, °C

Weight of the copper cylinder =

Height of the copper cylinder =

Diameter of the copper cylinder =

Radius of the copper cylinder =

Surface area of the copper cylinder =

REPORT:-

4. DETERMINATION OF RADIATION CONSTANT OF UNPAINTED GLASS

AIM:- To determine the radiation constant of un-painted glass.

REQUIREMENT:-

Round bottomed non painted flask

Beaker

Thermometer

Cork

Stand with clamp

Stop watch

PRINCIPLE:- Heat transfer by radiation involves the transfer of energy in the form of electromagnetic waves. All solid bodies radiate energy when their temperatures are above absolute zero. The principle form of radiant energy is thermal energy for industrial applications. The radiant energy emitted by a hot body is expressed by Stefan-Boltzmann law as given below:

$$q = bAT^4$$

Where, q = Energy radiated per second, W (or J/s)

A = Area of radiating surface, m^2

T = Absolute temperature of the radiating surface, K

B = Constant, $W/m^2.K^4$

The difference in the temperature of hot body and ambient is the temperature gradient for the heat loss by radiation. The radiation constant (α) is calculated using the following equation:

$$(M_1s_1 - M_2s_2) \frac{dq}{dt} = \alpha A [(T_1/100)^4 - (T_2/100)^4] + \beta A (T_1 - T_2)^{1.23}$$

Where,

M_1 = Mass of water, w g

M_2 = Mass of round bottom unpainted flask, kg

S_1 = Specific heat of the metal, J/Kg.K

S_2 = Specific heat of the glass, J/Kg.K

(dq/dt) = Rate of heat loss by metal cylinder, W/s

T_1 = Temperature of the metal body, K

T_2 = Temperature of the ambient (room temperature), K

α = Radiation constant, $W/m^2.K^4$

β = Convection factor

A = Surface area for heat transfer, m^2

PROCEDURE:-

1. Take a round bottom flask, measure the diameter, average radius and then surface area in determined whose heat loss to be calculated.
2. The Flask is hanged in air by tying one end for neck with a thread, and other end to a clamp of stand.
3. Boil the water upto its boiling point and taken in to the flask up to the neck level.
4. The flask is fitted with a rubber cork having one hole, which is fitted with thermometer.
5. The temperature is noted for every 5 min. till it reaches to room temperature.
6. A graph is plotted between temperature on Y-axis and time on X-axis.
7. Calculate radiation constant.

OBSERVATIONS AND CALCULATIONS:

Time, mins	Temperature, °C	Time, mins	Temperature, °C	Time, mins	Temperature, °C

Weight, M_1 =

Diameter of the flask, D =

Radius of the flask, R =

Diameter of the neck, d =

Radius of the neck,r =

Surface area,A =

Surface area of the iron cylinder =

REPORT:-

5. DETERMINATION OF RADIATION CONSTANT OF PAINTED GLASS

AIM:- To determine the radiation constant of painted glass.

REQUIREMENT:-

Round bottomed painted flask

Beaker

Thermometer

Cork

Stand with clamp

Stop watch

PRINCIPLE:- Heat transfer by radiation involves the transfer of energy in the form of electromagnetic waves. All solid bodies radiate energy when their temperatures are above absolute zero. The principle form of radiant energy is thermal energy for industrial applications. The radiant energy emitted by a hot body is expressed by Stefan-Boltzmann law as given below:

$$q = bAT^4$$

Where, q = Energy radiated per second, W (or J/s)

A = Area of radiating surface, m^2

T = Absolute temperature of the radiating surface, K

B = Constant, $W/m^2.K^4$

The difference in the temperature of hot body and ambient is the temperature gradient for the heat loss by radiation. The radiation constant (α) is calculated using the following equation:

$$(M_1s_1 - M_2s_2) \frac{dq}{dt} = \alpha A [(T_1/100)^4 - (T_2/100)^4] + \beta A (T_1 - T_2)^{1.23}$$

Where,

M_1 = Mass of water, w g

M_2 = Mass of round bottom unpainted flask, kg

S_1 = Specific heat of the metal, J/Kg.K

S_2 = Specific heat of the glass, J/Kg.K

(dq/dt) = Rate of heat loss by metal cylinder, W/s

T_1 = Temperature of the metal body, K

T_2 = Temperature of the ambient (room temperature), K

α = Radiation constant, $W/m^2.K^4$

β = Convection factor

A = Surface area for heat transfer, m^2

PROCEDURE:-

1. Take a round bottom flask, measure the diameter, average radius and then surface area in determined whose heat loss to be calculated.
2. The Flask neck is covered with a black carbon paper and is hanged in air by tying one end for neck with a thread, and other end to a clamp of stand.
3. Boil the water upto its boiling point and taken in to the flask up to the neck level.
4. The flask is fitted with a rubber cork having one hole, which is fitted with thermometer.
5. The temperature is noted for every 5 min. till it reaches to room temperature.
6. A graph is plotted between temperature on Y-axis and time on X-axis.
7. Calculate the radiation constant.

OBSERVATIONS AND CALCULATIONS:

Time, mins	Temperature, °C	Time, mins	Temperature, °C	Time, mins	Temperature, °C

Weight, M_1 =

Diameter of the flask, D =

Radius of the flask, R =

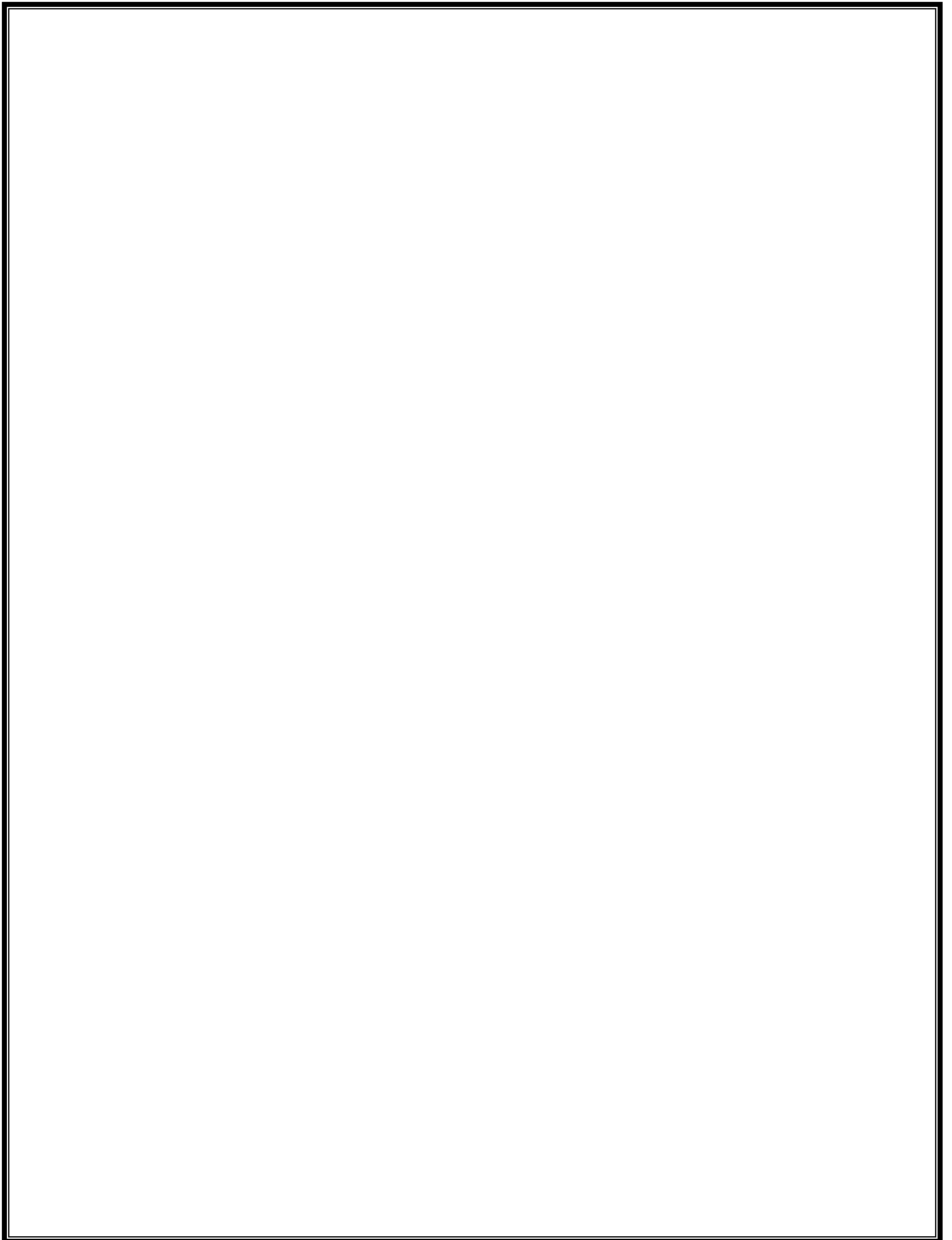
Diameter of the neck, d =

Radius of the neck,r =

Surface area,A =

Surface area of the iron cylinder =

REPORT:-



6. STEAM DISTILLATION- Separation of Turpentine Oil

AIM: To study the process of steam distillation.

PRINCIPLE: Steam distillation is a process of distillation carried with the aid of steam and is used to separate high-boiling substances from nonvolatile materials.

A mixture of immiscible liquids begins to boil when the sum of their vapour pressures is equal to the atmosphere pressure. In case of a mixture of water and turpentine oil, mixture boils below the boiling point of pure water, though turpentine boils at much higher temperature than that of water. The net result, high boiling substances may be distilled at a temperature much below its boiling point, when water (steam) is used. The turpentine oil is distilled along with water. These liquids are immiscible and separated using a separating funnel. Thus it is possible to separate and purify one liquid from a mixture.

APPLICATIONS:

- It is used for the separation of liquids immiscible with water, like toluene and water.
- This method is used for extracting volatile oils like clove, anise.
- It is useful in purification of liquid with high boiling point, ex- essential oil of almond.
- Aromatic waters are prepared by this method.

ADVANTAGES:

- Volatile oils can be separated at a lower temperature in steam distillation, without any decomposition and aroma.
- If a substance has low volatility, it can be satisfactorily distilled, provided its molecular weight is considerably higher than water.

DISADVANTAGES:

- Steam distillation is not suitable when immiscible liquid and water react with each other.

ASSEMBLY OF APPARATUS FOR STEAM DISTILLATION:

The assembly of apparatus for steam distillation on laboratory scale consists of a metallic (Copper) steam can fitted with a rubber cork having two holes. Through one of the hole, a long tube is passed so as to reach almost the bottom of the steam generator.

This tube acts as a safety tube, so that in case the pressure inside the steam generator becomes too much, water will be forced out of it and the pressure will be relieved. Moreover, when steam starts coming out from the safety tube; it indicates that the steam can is almost empty.

Through another hole, a bent tube is passed. The other end of the bent tube is connected to the flask containing non aqueous liquid through a rubber bung. This tube should reach almost the bottom of the flask.

Through another hole of the rubber bung, a delivery tube is inserted which connects the flask and the condenser. The condenser is connected to a receiver flask using an adaptor.

PROCEDURE:

Simple Distillation

- 50 ml of turpentine oil is placed in a 250 ml round bottom flask.
- The cork carrying thermometer is fitted to the flask. The tip (mercury) should be in front of the side tube of the flask.
- The flask is heated by Bunsen burner. The turpentine oil gets heated and after some time starts boiling.
- The temperature (T_1) is noted at which turpentine oil distills. This is boiling point of turpentine oil and remains constant.
- Simple distillation is continued to collect 25ml of condensate.

Steam Distillation

- 30 ml of turpentine oil is placed in 250 ml round bottom flask.
- 100 ml of water is added to the above flask
- The steam can (or round bottom flask) is filled with water and assemble the remaining.
- Both the steam can and flask are heated simultaneously, so that steam flows uniformly through the mixture.
- The mixture gets heated. After some time, it starts boiling.
- The temperature (T_2) at which boiling occurs is noted.

- Steam carries the vapour of oil and passes into the condenser where condensation takes place.
- Condensate is collected and oil is separated from water by using separating funnel.
- The weight of turpentine oil (w_2) and water (w_1) layers are noted.
- The percent efficiency is calculated and reported.

OBSERVATION AND CALCULATION:

Boiling point of turpentine oil by simple distillation, T_1 °C=

Boiling point of the mixture by steam distillation, T_2 °C=

Decrease in boiling point, $T_3 = T_1 - T_2 =$

Theoretical recovery ratio of turpentine oil to water=

Weight of water obtained, $w_1 =$

Weight of turpentine oil obtained, $w_2 =$

Practical recovery ratio of turpentine oil to water, $w_2/w_1 =$

Percent efficiency of steam distillation, $(\text{Practical recovery} / \text{Theoretical recovery}) \times 100 =$

REPORT:

7. DETERMINATION THE OVERALL HEAT TRANSFER COEFFICIENT BY HEAT EXCHANGER

AIM: -to determine the overall heat transfer coefficient by heat exchanger.

REQUIREMENT:-

Steam generator, Beaker, bent tube, water condenser, Thermometer,

PRINCIPLE: Heat transfer by convection is involved between two liquids, when these are separated by glass wall. The differences in the modes of feeding largely determine the efficiency of a heat process. Heat exchangers are the devices used for transferring heat from one fluid (hot gas or steam) to another fluid (liquid) through a metal wall. When the feed of hot fluid is passed through one end of the apparatus and the cold fluid is passed through the other end, this arrangement is known as counter-current or counter flow method. The overall heat transfer coefficient of a glass tube is mathematically expressed for a counter current flow as:

$$U = \frac{Q}{A \times \Delta t_{av}}$$

Where, Q = amount of heat transferred, W (J/s)

A = Surface area of the glass tube, m²

Δt_{av} = temperature gradient, K

U = overall heat transfer coefficient, W/ m²·K

In the above equation Q represented as:

$$Q = (Q_1 + Q_2) / 2$$

$$Q_1 = M_1 \cdot L + M_1 \cdot s \cdot \Delta t_1$$

$$Q_2 = M_2 \cdot s \cdot \Delta t_2$$

Where,

M_1 = Mass of condensed steam, kg

M_2 = mass of circulated water, kg

S = specific heat of steam, J/kg.K

L = latent heat of vaporization of water, J/kg

t_1 = temperature drop on steam, K

t_2 = temperature rise on the circulating water side, K

The temperature gradient, Δt_{av} is expressed as:

$$\Delta t_{av} = (\Delta t_1 + \Delta t_2)/2$$

Δt_1 = difference in temperature on steam side, K

Δt_2 = difference in temperature on cold water side, K

The water condenser used in the laboratory or distillation is an example of the counter current flow of liquids and heat transfer. Thus, overall heat transfer coefficient is determined using water condenser.

PROCEDURE:-

1. The length and diameter of the plain water condenser is determined.
2. Using the plain water condenser, the distillation apparatus is assembled.
3. The inlet of water condenser is connected to the tap. The outlet of the condenser is placed in the (2 lit) beaker.
4. The temperature of the tap water inlet is noted.
5. The steam generator is heated so that steam is produced. As the steam is generated, the steam thermometer shows constant temperature. This temperature is noted.
6. As the process continues, a steady state situation is obtained. At this stage, heat transfer measurements are made.
7. The condensate begins to collect into an empty beaker. At the same time, the water is collected from the outlet into an empty vessel.

8. After a lapse of time (i.e 5 or 10 or 15 mins) , collecting of condensate is stopped by removing the beaker from the condenser.
9. Exactly at the same time, collecting of tap water from the outlet is also stopped, by removing the bottle from the rubber tubing.
10. The condensate is swirled and the temperature is noted. The quantity of condensate is measured and recorded.
11. The tap water collected into the bottle is also swirled and temperature is noted. The quantity of the tap water is measured and recorded.

OBSERVATIONS AND CALCULATIONS:

Diameter of the condenser, $d=$

Radius of the condenser, $r=$

Length of the condenser, $l=$

Area of the condenser, $A=2\pi rl$

Latent heat of vaporization of water, $L= 226.1 \text{ J/kg}$

Specific heat of steam, $s= 4190 \text{ J/kg.K}$

Heat loss by steam, $Q_1=$

Heat gain by tap water, $Q_2=$

Heat transferred, $Q=$

	Steam temp. A	Tap water temp. (outlet) b	Condensate temp. c	Tap water temp. (inlet),d
Temperature, °C				
Temperature, K				
Difference in temperatures	$\Delta t_1 = (a-b) =$		$\Delta t_2 = (c-d) =$	
Average temperature, Δt_{av}	$(\Delta t_1 + \Delta t_2)/2$			

REPORT:

8. CONSTRUCTION OF DRYING CURVE(Calcium carbonate)

AIM:- To dry calcium carbonate slurry and plot the rate of drying curves.

REQUIREMENTS:- Calcium carbonate, hot air oven, balance, petri plate, beaker etc.

PRINCIPLE:- Drying is defined as removal of small amounts of water or other liquid from a material by application of heat. Drying rate relationship can be studied considering a simple model which mimics the conditions of a dryer. In this model, the wet slab to be dried is placed in a tray whose bottom end sides are insulated. The air is blown over the solid under the constant drying conditions (like temperature, humidity, pressure etc). the superficial water diffuses through the surrounding air film and is carried away rapidly by the moving air stream. Then water diffuses from the interior of the solid to the surface. This process continues until bound water gets evaporated. Then the material attains equilibrium moisture content.

Rate of drying of this process can be determined by periodically weighing the calcium carbonate slurry. The difference in the weights of two successive weighing gives the loss of moisture content, i.e., amount dried. The following equation is used to calculate rate of drying:

$$\text{Rate of Drying} = \frac{\text{Weight of water removed}}{\text{Weight of dry powder X time of drying} \times \text{X surface area exposed}} \quad \text{g/g.h.cm}^2$$

PROCEDURE:-

1. The petri plate is weighed and the weight is recorded as W_1 .
2. 15.0 g of calcium carbonate is transferred into a beaker. Water (about 30 ml) is added slowly to prepare slurry.
3. The calcium carbonate slurry is transferred into the petri plate.
4. Filling must be done in such a way that $3/4^{\text{th}}$ of the volume of the stainless steel plate is filled with slurry.
5. The weight of petri plate plus slurry is taken and recorded as W_2 .
6. The plate containing slurry is placed in hot air oven, whose temperature must be maintained at 60°C .
7. The time is noted soon after placing plate containing slurry in hot air oven.
8. After 15 mins, the wt. of plate with slurry is taken. The weight is recorded in table.

9. Again the petri plate containing slurry is placed in the dryer. (petri plate should be immediately placed back into the dryer , otherwise temperature decreases enormously and results will be erroneous.
10. Step 8 and 9 are repeated until constant weight is obtained.
11. The rate of drying is calculated.
12. A graph is plotted by taking free moisture content (weight of water) on x-axis and rate of drying on y-axis.

OBSERVATION AND CALCULATIONS:-

S N	Time, mins	Wt. of empty petridish (W ₁) gm	Wt. of petridish + CaCO ₃ (W ₂) gm	Wt. of petridish + CaCO ₃ + Water (W ₃) gm	Wt. of petridish + CaCO ₃ + Water after drying (W ₄) gm	Moisture evaporate d in 15 mins time interval (W ₃ - W ₄)	Total moisture content (W ₄ - W ₂)	Moisture content on drying basis (W ₄ - W ₂)/ (W ₂ - W ₁)	Average moisture content	Rate of drying gm/min/cm ² (W ₃ - W ₄) / Time X Area
1										
2										
3										
4										
5										
6										
7										
8										
9										

1. Wt. of empty petridish, W₁ =
2. Wt. of petridish + CaCO₃, W₂ =
3. Wt. of petridish + CaCO₃ + Water ,W₃ = W₂ - W₁ =
4. Diameter of petridish, d = cm

5. Radius of petridish, $r = d/2 =$ cm
6. Area of petridish, $A = \pi r^2 =$ cm²
7. Moisture content at '0' time =
8. Moisture content at '15' mins =
9. Average moisture content =

REPORT:-

9. CONSTRUCTION OF DRYING CURVE(Starch)

AIM:- To dry starch slurry and plot the rate of drying curves.

REQUIREMENTS:- starch, hot air oven, balance, petri plate, beaker etc.

PRINCIPLE:- Drying is defined as removal of small amounts of water or other liquid from a material by application of heat. Drying rate relationship can be studied considering a simple model which mimics the conditions of a dryer. In this model, the wet slab to be dried is placed in a tray whose bottom end sides are insulated. The air is blown over the solid under the constant drying conditions (like temperature, humidity, pressure etc). the superficial water diffuses through the surrounding air film and is carried away rapidly by the moving air stream. Then water diffuses from the interior of the solid to the surface. This process continues until bound water gets evaporated. Then the material attains equilibrium moisture content.

Rate of drying of this process can be determined by periodically weighing the calcium carbonate slurry. The difference in the weights of two successive weighing gives the loss of moisture content, i.e., amount dried. The following equation is used to calculate rate of drying:

$$\text{Rate of Drying} = \frac{\text{Weight of water removed}}{\text{Weight of dry powder X time of drying} \times \text{X surface area exposed}} \quad \text{g/g.h.cm}^2$$

PROCEDURE:-

1. The petri plate is weighed and the weight is recorded as W_1 .
2. 15.0 g of starch is transferred into a beaker. Water (about 30 ml) is added slowly to prepare slurry.
3. The starch slurry is transferred into the petri plate.
4. Filling must be done in such a way that $3/4^{\text{th}}$ of the volume of the stainless steel plate is filled with slurry.
5. The weight of petri plate plus slurry is taken and recorded as W_2 .
6. The plate containing slurry is placed in hot air oven, whose temperature must be maintained at 60°C .
7. The time is noted soon after placing plate containing slurry in hot air oven.
8. After 15 mins, the wt. of plate with slurry is taken. The weight is recorded in table.
9. Again the petri plate containing slurry is placed in the dryer. (petri plate should be immediately placed back into the dryer, otherwise temperature decreases enormously and results will be erroneous).
10. Step 8 and 9 are repeated until constant weight is obtained.
11. The rate of drying is calculated.
12. A graph is plotted by taking free moisture content (weight of water) on x-axis and rate of drying on y-axis.

OBSERVATION AND CALCULATIONS:-

S N	Time, mins	Wt. of empty petridish (W ₁) gm	Wt. of petridish + CaCO ₃ (W ₂) gm	Wt. of petridish + starch + Water (W ₃) gm	Wt. of petridish + starch + Water after drying (W ₄) gm	Moisture evaporate d in 15 mins time interval (W ₃ - W ₄)	Total moisture content (W ₄ - W ₂)	Moisture content on drying basis (W ₄ - W ₂)/ (W ₂ - W ₁)	Average moisture content	Rate of drying gm/min/cm ² (W ₃ - W ₄) / Time X Area
1										
2										
3										
4										
5										
6										
7										
8										
9										

1. Wt. of empty petridish, W₁ =
2. Wt. of petridish + starch , W₂ =
3. Wt. of petridish + starch + Water , W₃ = W₂ - W₁ =
4. Diameter of petridish, d = cm
5. Radius of petridish, r = d/2 = cm
6. Area of petridish, A = πr² = cm²
7. Moisture content at '0' time =
8. Moisture content at '15' mins =
9. Average moisture content =

REPORT:-

10. DETERMINATION OF HUMIDITY OF AIR BY DEW POINT METHOD

AIM: -To determine humidity of Air by using Dew point method.

REQUIREMENT:-

Round bottom flask having polished surface.

Thermometer

Tripod Stand

Stirrer

Humidity Chart

PRINCIPLE: - The **Dew point temperature** (DPT) is the temperature to which a mixture of air-water vapour must be cool (at constant pressure and constant water vapor content) in order to reach saturation. Formation of mist and disappearance of mist are considered and dew point is determined. Dew point temperature is noted on the temperature axis (x-axis) and moved vertically on the psychrometric chart. The intersect point at saturated curve (100%) is identified. The coordinates of the point (temperature, K, humidity) are noted. The y-axis point is the humidity of air. These values are substituted in the equation:

Percent relative humidity= (humidity of air/ humidity of saturated air) X 100

PROCEDURE:-

1. Take a polished round bottom flask (100 ml) and fill water upto 2/3 of its volume
2. Place the flask on tripod stand and fix it.
3. Hang a thermometer hanging from the main stand such that the thermometer's bulb is dipping into the water in the vessel.
4. Drop small pieces of ice cubes into the vessel one by one slowly, under continuous stirring of the water with the help of glass rod or magnetic stirrer. Continue the stirring until a film of moisture (mist) is formed on the polished surface of flask.
5. Note the temperature of this stage which is dew point and record it.
6. Denote humidity of air by using dew point with the help of humidity.

OBSERVATION AND CALCULATION:-

Trails	Dew point, °C		Humidity
	Mist appearance	Average value	

From humidity chart, humidity of saturated air dew point =

Percent relative humidity =

REPORT:-

11. DETERMINATION OF HUMIDITY OF AIR BY WET AND DRY BULB TEMPERATURE

AIM: -To determine humidity of air by wet and dry bulb temperature.

REQUIREMENT:-

Thermometer

Humidity Chart

PRINCIPLE: - **Humidity** is defined as the amount of water vapor present in a unit volume of air, usually expressed in kilograms per cubic meter. It can also be defined as the ratio of mass of water present in the air to the mass of dry air. **Relative humidity** is defined as the ratio of actual humidity to the saturation humidity at a temperature. The **dry-bulb temperature (DBT)** is the temperature of air measured by a thermometer freely exposed to the air but shielded from radiation and moisture. Temperature is usually measured in degrees Celsius ($^{\circ}\text{C}$), Kelvin (K), or Fahrenheit ($^{\circ}\text{F}$). The **Wet bulb temperature (WBT)** is the temperature of air measured by a thermometer having a wick moistened with distilled water. Temperature is usually measured in degrees Celsius ($^{\circ}\text{C}$), Kelvin (K), or Fahrenheit ($^{\circ}\text{F}$). Wet-bulb temperature is largely determined by both actual air temperature (dry-bulb temperature) and the amount of moisture in the air (humidity). At 100% relative humidity, the wet-bulb temperature equals the dry-bulb temperature.

PROCEDURE: - Dry bulb temperature is the temperature, which we will get, if a thermometer is placed in a sample of air. In a sample of air, if we place the thermometer, then we shall see somewhere there is the mercury level and that gives the temperature of the sample. So that is dry bulb temperature or simply we can call it temperature of the sample. For wet bulb temperature we have a similar thermometer but the bulb of this thermometer is wrapped with the help of a layer of cotton and then, this cotton is kept moist with the help of distilled water. We shall get two readings- one from the dry bulb thermometer and another from wet bulb thermometer. These readings are used for finding humidity from adiabatic cooling line in the psychrometric charts.

OBSERVATIONS AND CALCULATIONS:-

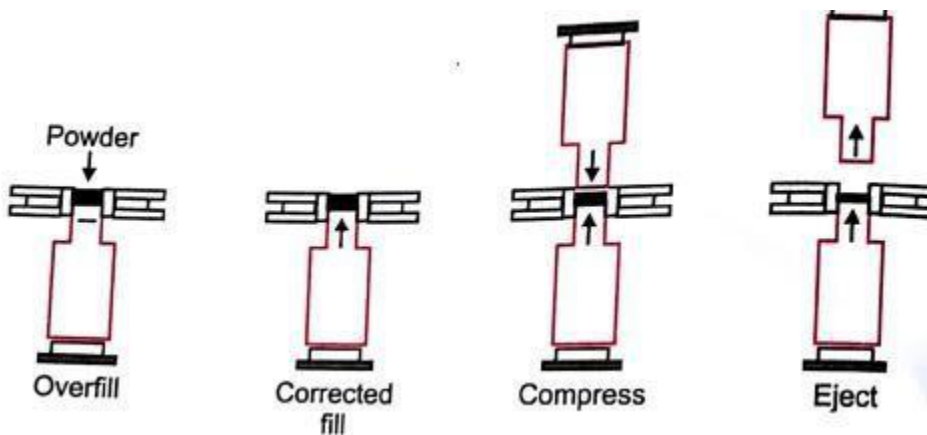
Average dry bulb temperature		Average dry bulb temperature		Humidity	Percent relative humidity
°C	°F	°C	°F		

REPORT:-

12. DESCRIPTION OF CONSTRUCTION, WORKING AND APPLICATION OF PHARMACEUTICAL MACHINERY (ROTARY TABLET MACHINE, FLUIDIZED BED COATER, FLUID ENERGY MILL, DE HUMIDIFIER)

I. ROTARY TABLET MACHINE

It is also called multi station tablet press. It is called rotary machine because the head of machine that holds the upper punches, dies and lower punches in places rotates. Steps involved in manufacturing of tablet.



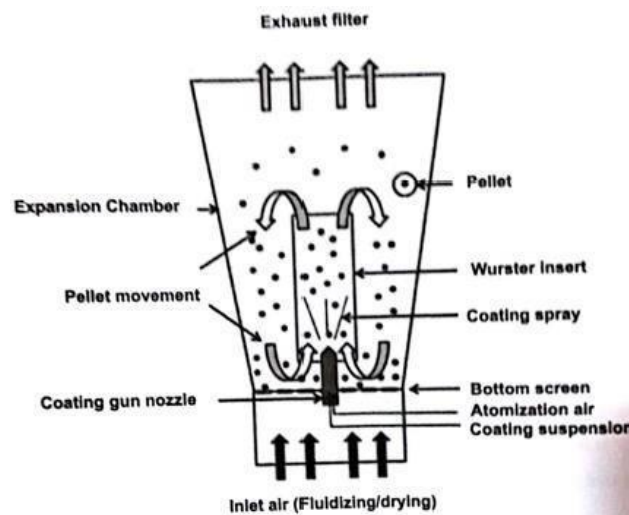
- The material to feed through hopper.
- The fill cam pulls the lower punches down to affixed distance and the dies are filled with material.
- The quantity of the material filled is larger than the actual amount required. Remove excess amount with the help of Spatula.
- After that, upper punch is lower and inserted into the dies.
- The material is compressed and the tablets are formed.
- After the compression, pulls the upper punches into their top position and simultaneously lifts the lower punches until the tablets are ejected from the dies.
- Then tablet is passed through the discharge chute.

APPLICATIONS

- It is operated continuously
- Used for large scale production
- A single rotary press produce 1150 tablets in a minute while double rotary press can produce 10,000 tablets in a minute.

II. FLUIDIZED BED COATER:

Three types of air suspension coater are available, namely, top spray coater, wurster or bottom spray coater and tangential spray coater. In top spray coater, there is a counter (opposite direction) movement of powder particles or pellets and liquid spray. In wurster or bottom spray coater, there is a concurrent (same direction) movement of powder particles or pellets and liquids spray in tangential spray coater the powder particles or pellets move in a helical fashion due to spinning rotor disk on the bottom of the equipment. Steps involved in wurster or bottom spray coater



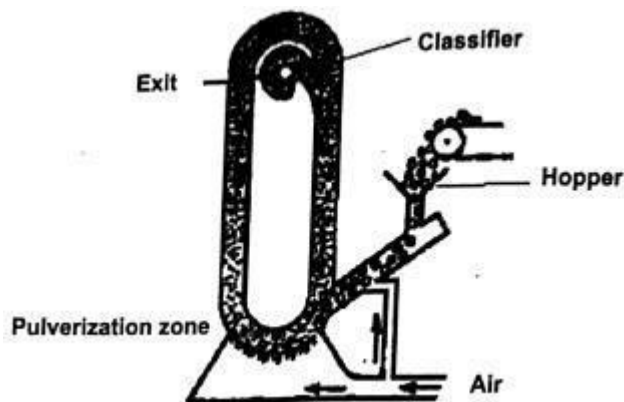
- The drying inlet air is passed upwards through the bottom perforated plate into the fluid bed chamber.
- This air passes to wurster column, in which a spray gun perpendicular to bottom plate and parallel to the wurster column.
- The air is passed out from the exhaust filters situated at the top of the equipment.
- The material to be coated is loaded in the fluid bed chamber and fluidized.

- The inlet air cause fluidization of the material as well as its drying during the coating operation.
- The pellets are passing through the liquid spray of coating solution from the spray gun positioners parallel to the column.
- After coating the coated particles falls by gravity at the bottom of wurster column and recycled to coating zone.

APPLICATIONS:

- It is used to coat pharmaceutical dosage form with polymeric material to mask objectionable taste or odor and also to protect an unstable ingredient and to improve appearance.
- Fluidized bed coaters are used for coating of powders, granules, tablets, pellets etc by column of air.
- Fluid bed coating equipment is popular for coating multi particulate systems such as beads and non-parcel seeds.

III. FLUID ENERGY MILL

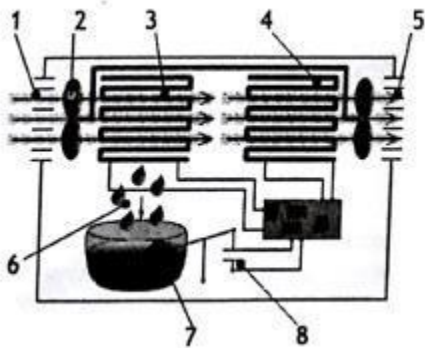


- A fluid usually air, is injected at very high pressure through nozzles at bottom of the loop. As a results turbulence produce.
- Solids are introduced into the stream through hopper.
- Due to this turbulence occur and impacts and attrition occur between the particles.
- A classifier is fitted at the exist so that only finer size particles are collected as products.
- The larger size particles are again sent to the stream of air for futher size reduction.

APPLICATIONS:

- The particle size of the product is smaller when compared to other method of size reduction.
- No chance of contamination of the product.
- This method is suitable where fine powders are required like micronization of griseofulvin.

DEHUMIDIFIER



- Warm moist air is sucked in through one side of the machine.
- An electric fan is used to draw the air inward.
- The warm air passes through cold pipes through which a coolant circulates. Due to cooling of air the moisture it contains turns back into liquid water.
- Then the air passes over a heating element and warms back up to its original temperature.
- Warm dry air blows back into the room through another side of machine.
- The moisture that was in the air drips down into collecting tray (or bucket) at the bottom of the machine.
- As the collecting tray fills up a plastic float in the machine rises upward.
- When the tray is full the float trips an electric switch that turns off the fan and switches on an indicator light which indicates that the machine needs emptying.

APPLICATIONS

- A dehumidifier is used to reduce the level of humidity in the air.
- Large dehumidifiers are also used in commercial buildings such as indoor ice rinks to control the humidity level

13 SIZE ANALYSIS BY SIEVING

AIM:-To determine the particle size distribution of powder by sieving method.

REQUIREMENTS:-

Sieve set (sieve no. 30, 45, 60, 100, 140, and 200), Electromagnetic laboratory sieve machine or electrical sieve shaker

CHEMICAL / REAGENTS:-

Calcium carbonate/aspirin/ calamine powder/any power substances

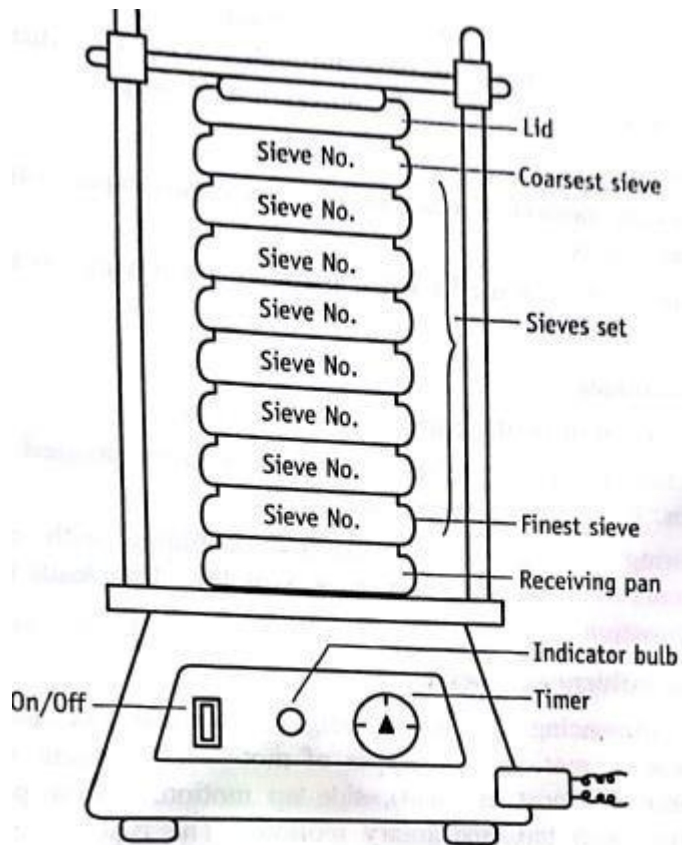
PRINCIPLE:

The basic principle involved in this method is size separation using standard sieves or screens

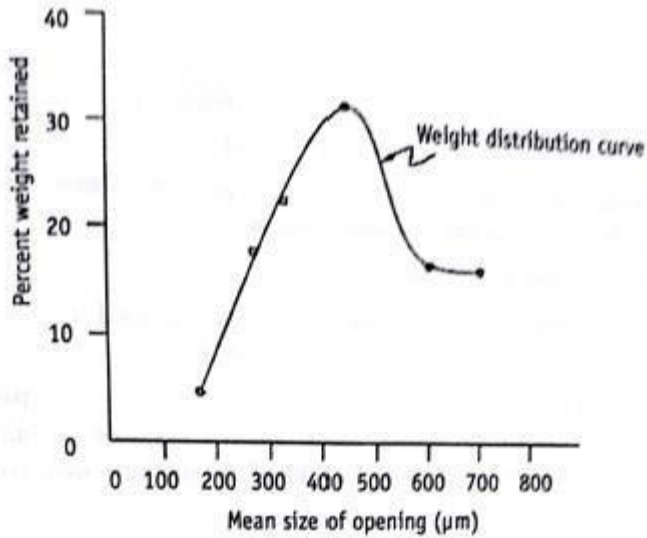
Size separation is unit operation that involves the separation of various sizes of particles into two or more portions by means of screening surfaces. size separation is also known as sieving, sifting, classifying or screening.

Designations and dimensions of IP and USP specification sieves

Sieve number		Nominal mesh aperture size, mm		Sieve number		Nominal mesh aperture size, μm	
IP	USP	IP	USP	IP	USP	IP	USP
4	8	4000	2380	36	50	425	297
8	10	2000	2000	44	60	355	250
10	20	1700	840	60	70	250	210
12	25	1400	710	85	80	180	177
16	30	1000	595	100	100	150	149
22	35	710	500	120	120	125	125
25	40	600	420	150	140	106	105
30	45	500	350				



The powdered drug is separated according to its particle size using a number of sieves in a nest. These are subjected to different types of agitation in sieve shaker, so that size separation is rapid. Sieves are arranged in a nest with the coarsest at the top. A sample of the powder or granules is placed on the top sieve. This sieve set is fitted to the mechanical gyratory shaker and shaken for a period of time. The powder retained on each sieve is weighed. Then normal weight distribution curve is constructed.



The average particle diameter of a powder (d_{sieve}) is calculated using the following equation.

$$d_{sieve} = \frac{\sum(n \times d)}{\sum(n)}$$

Where n = frequency of particles in a particle size range, g; or percent weight of powder
undersize, g

d = average particle diameter of particular sieve (sieve diameter), μm

PROCEDURE:-

1. Arrange set of sieves in the descending order.
2. Weighed amount of sample is tube placed in the sieve at the top of the sieve set.
3. Start the sieving machine. The length of time and speed of vibration can be controlled by semiautomatic attachment in the machine.
4. Collect the powder material retained on the various sieves.
5. Weigh the powder material retained on the sieves.
6. Calculate percent frequency of each size of particle and plot the graphs.
7. Determine the geometric mean weight diameter and geometrical standard deviation.

OBSERVATIONS :-

- (a) Weight of substance, W_1 = g
- (b) Time of shaking = min
- (c) Speed of electrical shaker = rpm

S. no.	Sieve number (passed/retained)	Arithmetic Mean size Of opening (μm)	Weight Retained on a sieve (g)	Percent Weight Retained (undersize)	Cumulative Percent Retained
1	30/45	470			
2	45/60	300			
3	60/80	213			
4	80/100	163			
5	100/140	127			
6	140/200	90			

CALCULATION :-

Calculation of percent weight retained on screen.

Weight retained on screen = 100

Percent weight retained on screen = $\frac{\text{Total weight of powder}}{\text{Total weight of powder}} \times 100$

1. Plot frequency distribution curve taking particle size on X axis and percent weight retained on the screen on Y axis.
2. The logarithm of the particle size is plotted against the cumulative percent frequency on a probability scale. It showed a linear relationship.

3. The geometrical mean weight diameter d_g and geometrical standard deviation can be obtained from the straight line.

REPORT:

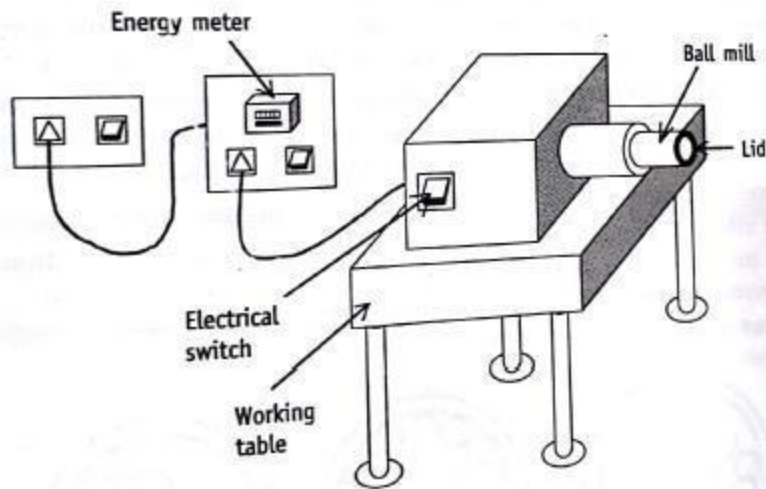
14. SIZE REDUCTION BY BALL MILL

AIM: Size reduction by ball mill and to verify the laws of size reduction using ball mill and determining Kicks, Rittinger's, Bond's coefficients, power requirement and critical speed of Ball Mill.

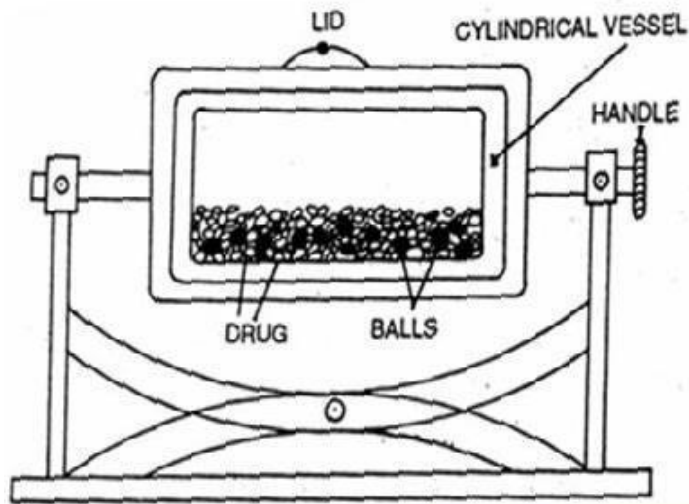
REQUIREMENTS: Ball mill, energy meter, granules, balance, sieves, sieve shaker.

PRINCIPLE:

Size reduction is a process of reducing large solid masses (vegetable and chemical substances) into small unit masses, coarse particles or fine particles.



Ball mill with electric circuit



Ball mill

Ball mill are also known as tumbling mills or pebble mills. The ball mill works on the principle of impact between the rapidly moving balls and the material. It consists of a hollow cylinder with metal balls acting as a grinding medium. Balls occupy 30-50% of the total volume. The hollow cylinder rotates around the longitudinal axis for the size reduction of material placed inside it. A ball mill is used especially for reducing the properties of both wet and dry powder, which can produce desired results. The speed with which the mill rotates should be optimum. At this speed, the ball will fall and strike the bottom of the mill, crushing the powder into small pieces. This produces an impact stress on the material to be ground.



(A) Balls at low speed



(B) Balls at high speed



(C) Balls at critical speed

KICK'S LAW:

It states that the energy required for size reduction is proportional to the logarithm of the ratio between the initial and final size. For crushing (compression) of large particles, Kick's equation is more useful.

$$E = K_K \ln \left(\frac{d_i}{d_n} \right)$$

Where, E= amount of energy required to produce a change in unit mass,

K_K =Kick's constant energy per unit mass

d_i =initial particle size of sample (before size reduction), μm .

d_n = final particle size of sample (after size reduction), μm .

RITTINGER'S LAW:

It states that the energy consumed in the size reduction of solids is directly proportional to the new surface created. It is mostly applicable to brittle materials undergoing fine milling

$$E = K_R \left(\frac{1}{d_n} - \frac{1}{d_i} \right)$$

Where E=amount of energy required to produce a change in unit mass.

K_r =Rittinger's constant energy per unit area.

BOND'S WORK INDEX:

It states that the energy used for deforming a set of particles of equivalent shape is proportional to the change in particle dimensions. The bond's work index is a useful way for comparing the efficiency of milling operation. This is useful for rough mill sizing.

$$E = 2K_B \left(\frac{1}{\sqrt{d_n}} - \frac{1}{\sqrt{d_i}} \right)$$

Where, K_B =Bond's work index, energy per unit mass

PROCEDURE:

1. The initial dial reading of energy meter is noted as N_1 .
2. The cleaned ball mill is taken with sufficient number of balls.
3. The ball mill is operated without load for 10 min.
4. The reading (revolution) in energy meter is noted down as N_2 , (The difference, i.e., $N_3 = N_2 - N_1$ gives the energy required for running the ball mill without feed).
5. Hundred grams of sample is weighed and subjected to sieve analysis. The average particle size of the sample is calculated.
6. Hundred grams of feed, which was subjected to sieve analysis is transferred into the ball mill.
7. The ball mill is operated for 10 minutes.
8. The reading (revolutions) is noted as N_4 . (The difference, i.e., $N_5 = N_4 - N_2$ gives the energy required for running the ball mill and size reduction of material).
9. The difference i.e., $N_6 = N_5 - N_3$, gives the energy actually consumed for the size reduction of material.
10. The product is unloaded on to a tray and subjected for sieve analysis.
11. The average particle size of the product after size reduction is determined.

12. The data is substituted in equations of Kick's constant, Rittinger's constant, and Bond's work index to determine them.

OBSERVATIONS AND CALCULATIONS:-

Table No: Weight distribution of sample after size reduction.

S. No.	Sieve No.	Nominal mesh aperture size, μm	Aperture Size(passed/retained), μm	Mean size of opening* (d), μm	Weight of powder undersize (n), gm	Percent weight retained on smaller sieve, (d)	Weight size n x d (4) x (6)
	(1)	(2)	(3)	(4)	(5)	(6)	(7)
	Pan		-	-	-	-	-
1	120	125	125/Pan	125.0			
2	100	150	150/125	137.5			
3	85	180	180/150	165			
4	60	250	250/180	215			
5	44	355	355/250	302.5			
6	22	840	710/355	532.5			
7	10	1700	1700/710	1205.5			
						$\Sigma(n) =$	$\Sigma(nd) =$

Mean diameter of the powder sample before size reduction, $d_i = \Sigma nd / \Sigma n$

CALCULATIONS:

1. Initial reading of energy meter, $N_1 =$
2. Energy meter reading after the use of ball mill without feed, $N_2 =$
3. Energy consumed by the ball mill, $N_3 = N_2 - N_1$
4. Energy meter reading after the use of ball mill with feed, $N_4 =$
5. Energy consumed for size reduction plus the ball mill, $N_5 = N_4 - N_2$
6. Energy consumed for the size reduction, $N_6 = N_5 - N_3 =$
7. Weight of the sample taken, $w = 100 \text{ g (0.1 kg)}$.

8. Calculation of energy meter constant , E (on the energy meter, the relationship between the revolution and energy is given. Use that relationship for calculations).

750 units reading (revolutions) the energy = 3600 kW.s (1 kW.h)

1 unit reading

$$1 \text{ unit reading} = 3600/750 = 4.8 \text{ kJ (E)}$$

9. Energy on no load, $E_1 = N_3 \times E =$ kJ.

10. Energy on load, $E_2 = N_5 \times E =$ kJ.

11. Net energy required per unit mass = $\{(E_2 - E_1)/w\} =$ kJ.

12. Average particle diameter of feed, $d_i =$ μm .

13. Average particle diameter of product, $d_n =$ μm .

REPORT: Rittinger's constant =

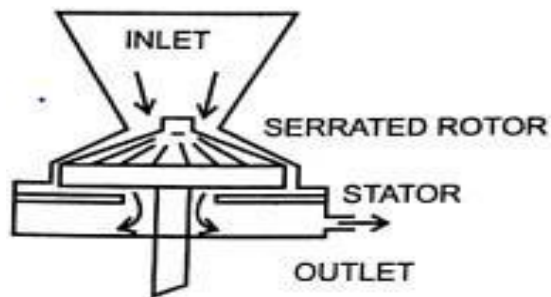
Kick's constant =

Bond's work index =

14.DEMONSTRATION OF COLLOID MILL, PLANETARY MIXER, **FLUIDIZED**

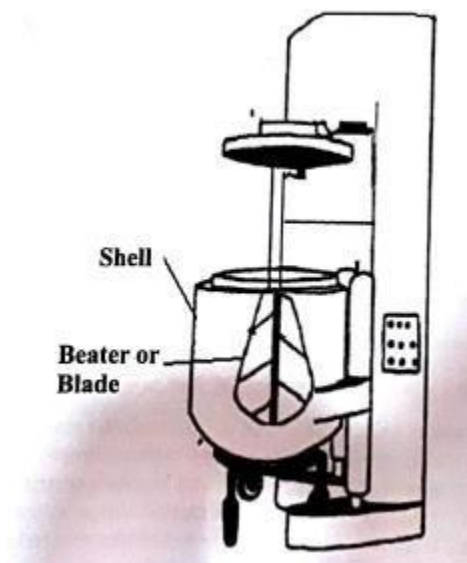
BED DRYER, FREEZE DRYER

COLLOID MILL:



- The colloid mill used to reduce the size of the suspended droplets.
- The materials is feed in though the inlet hopper and placed into the mill
- It is then move through the narrow gap between the rotor and stator to reduced the particle size
- Then final product is removed through the outlet.

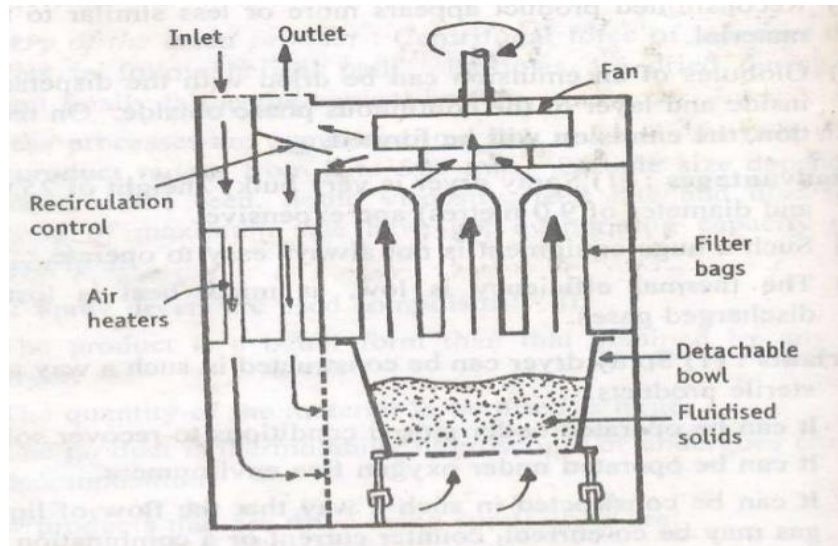
PLANETARY MIXER:



- The material to be mixed is loaded into mixing bowl or shell.

- The blades rotate on their own axis when they orbit the mixing bowl on a common axis. Therefore there is no dead spot in the mixing and high shear is applied for mixing.
- After mixing, the material is discharged through a bottom valve, or by manual scooping of the material from the bowl.

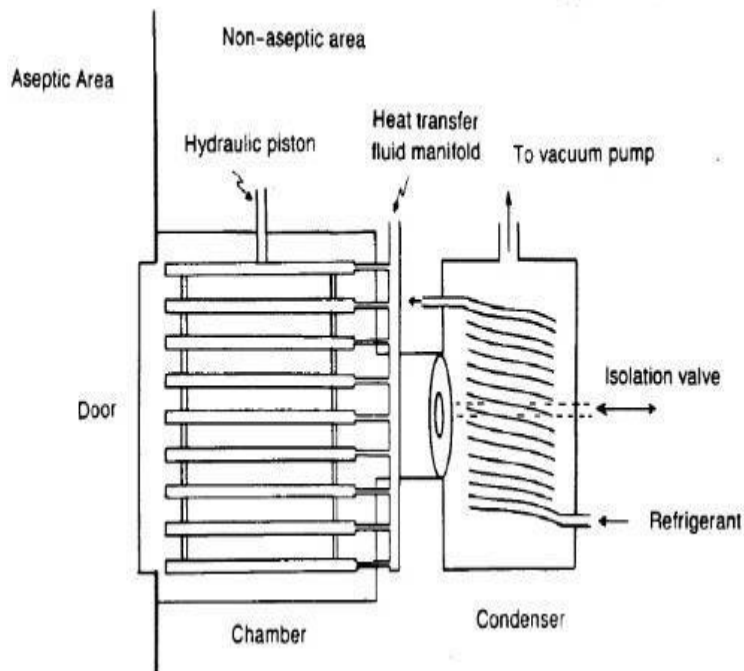
FLUIDIZED BED DRYER:



- The wet granules to be dried are placed in a detachable bowl. The bowl is inserted in the dryer.
- Fresh air can pass through a pre-filter, which is then heated when passing through a heat exchanger.
- Hot air flows through the bottom of the bowl. At the same time, the fan starts to rotate. The air speed increases gradually.
- After a specific times, a pressure point is reached in which the friction drag on the particles is equal to the force of gravity. The granules rise in the container. This condition is said to be fluidized.
- The gas surrounds each granule to dry them completely. The air comes out of the dryer passing through the filters in the bag.

- The entrained particles remain adhered to the interior of the surface of the bags.
Periodically, the bags are shaken to remove entrained particles.
- The materials are left in the dryer to reach room temperature.
- The bowl is removed for unloading. The final product is free flowing.

FREEZE DRYER:



- The material is pretreated before freezing pretreatment methods include freezing concentration, solution phase concentration, formulation to preserve the appearance of the product, formulation to stabilize reactive products, formulation to increase the surface area and decreasing high vapor pressure solvent.
- The product should be frozen at a temperature low enough to solidify completely. The products are frozen in two ways, most of the products that are lyophilized consist mainly of water. It is very important in lyophilization process.

- After pre-freezing the product, conditions must be established in which the ice can be removed from the frozen product through sublimation, resulting in a dry, structurally intact product.
- After primary freeze-drying is complete, and all ice has sublimated, bound moisture is still present in the product. The product appears dry, but the residual moisture content may be as high as 7-8% continued drying is necessary at warmer temperature to reduce the residual moisture content to optimum values. This process is called 'isothermal desorption'.
- After vacuum is replaced by inert gas, bottle and vials are closed.

15.DETERMINATION OF EFFECT OF CONCENTRATION, SURFACE AREA AND THICKNESS ON RATE OF FILTRATION

AIM: To determine the influence of concentration, surface area and thickness of filter medium on rate of filtration.

REQUIREMENTS: Measuring cylinder, Buckner funnel, filter paper, stop watch, calcium carbonate

PRINCIPLE: - Filtration may be defined as a process of separation of solids from a fluid by passing the same through a porous medium that retains the solids, but allows the fluid to pass through.

The rate of filtration depends on the concentration of solids. As the concentration of solids in the suspension increases, the thickness of the filter cake increases. As a result, rate of filtration decreases. The rate of filtration is directly proportional to surface area of the filter medium. Filter medium of sufficient thickness is used for filtration. But the thickness of the filter medium plays important role in determining the rate of filtration. Sometimes, increase in thickness decreases the rate of filtration. The rate of filtration also depends on the thickness of the filter cake formed.

The rate of filtration for practical purpose is calculated using the following equation:

$$\text{Rate of filtration} = \frac{\text{Volume of filtrate passed through the filter medium}}{\text{Time required for the filtrate to pass through}}$$
$$= \text{m}^3/\text{sec}$$

PROCEDURE:

EFFECT OF CONCENTRATION

Preparation of calcium carbonate suspension (5%)

g of calcium carbonate is weighed and transferred to the mortar. 25 ml of water is added and triturated to get smooth paste. The contents are transferred to measuring cylinder (50 ml). The mortar and pestle are washed with 5 ml of water (2 to 3 times if necessary). The washings are transferred to measuring cylinder (50 ml) and make up to mark with water. The suspension is shaken thoroughly.

The same procedure is repeated for 10% and 15% suspensions using 10.0 and 15.0 g of calcium carbonate, respectively.

Method for studying the effect of concentration

1. The filter paper of appropriate size is placed into a Buckner funnel.
2. 50 ml of 5% calcium carbonate suspension is poured over the Buckner funnel.
3. Time required to collect 25 ml of the filtrate is recorded.
4. The experiment (step 2 to 4) is repeated for the same conc. of calcium carbonate suspension for two more trials.
5. The experiment is repeated for other conc. (10% and 15%)also.
6. A graph is plotted by taking conc. of calcium carbonate on x-axis and rate of filtration on y-axis.

OBSERVATIONS AND CALCULATIONS:

Conc. Of slurry	Trial	Volume of filtrate collected (ml)	Time of collection of filtrate (Min)	Time of collection of filtrate (Sec)	Rate of filtration, ml/sec	Rate of filtration, m ³ /sec
1	2	3	4	5	6 (3)/(5)	7 (6)X 10 ⁻⁶
5%	1	25				
	2	25				
	Mean	25				
10%	1	25				
	2	25				
	Mean	25				
15%	1	25				
	2	25				
	Mean	25				

EFFECT OF SURFACE AREA

Preparation of calcium carbonate suspension (5%):

g of calcium carbonate is weighed and transferred to the mortar. 25 ml of water is added and triturated to get smooth paste. The contents are transferred to measuring cylinder (50 ml). The mortar and pestle are washed with 5 ml of water (2 to 3 times if necessary). The washings are transferred to measuring cylinder (50 ml) and make up to mark with water. The suspension is shaken thoroughly.

Method for studying the effect of surface area on rate of filtration

1. The filter paper of appropriate size is placed into a Buckner funnel.
2. 50 ml of 5% calcium carbonate suspension is poured over the Buckner funnel.
3. Time required to collect 25 ml of the filtrate is recorded.
4. The experiment (step 2 to 4) is repeated for the same conc. of calcium carbonate suspension for two more trials.
5. The experiment is repeated for medium and bigger Buckner funnel also.
6. A graph is plotted by taking surface area on x-axis and rate of filtration on y-axis.

OBSERVATIONS AND CALCULATIONS:

Surface area of filter medium, m ²	Trial	Volume of filtrate collected (ml)	Time of collection of filtrate (Min)	Time of collection of filtrate (Sec)	Rate of filtration, ml/sec	Rate of filtration, m ³ /sec
1	2	3	4	5	6 (3)/(5)	7 (6)X 10 ⁻⁶
Small	1	25				
	2	25				
	Mean	25				
Medium	1	25				
	2	25				
	Mean	25				
Big	1	25				
	2	25				
	Mean	25				

EFFECT OF THICKNESS OF FILTER MEDIUM

Preparation of calcium carbonate suspension (5%):

g of calcium carbonate is weighed and transferred to the mortar. 25 ml of water is added and triturated to get smooth paste. The contents are transferred to measuring cylinder (50 ml). The mortar and pestle are washed with 5 ml of water (2 to 3 times if necessary). The washings are transferred to measuring cylinder (50 ml) and make up to mark with water. The suspension is shaken thoroughly.

Method for studying the effect of thickness of filter medium on rate of filtration

1. The filter paper of known thickness (one filter paper) is placed into a Buckner funnel.
2. 50 ml of 5% calcium carbonate suspension is poured over the Buckner funnel.
3. Time required to collect 25 ml of the filtrate is recorded.
4. The experiment (step 2 to 4) is repeated for the same conc. of calcium carbonate suspension for two more trials.
5. The experiment is repeated by taking two and three filter papers.
6. A graph is plotted by taking thickness of filter medium on x-axis and rate of filtration on y-axis.

OBSERVATIONS AND CALCULATIONS:

Thickness of filter medium	Trial	Volume of filtrate collected (ml)	Time of collection of filtrate (Min)	Time of collection of filtrate (Sec)	Rate of filtration, ml/sec	Rate of filtration, m ³ /sec
1	2	3	4	5	6 (3)/(5)	7 (6)X 10 ⁻⁶
1 filter paper	1	25				
	2	25				
	Mean	25				
2 filter paper	1	25				
	2	25				
	Mean	25				
3 filter paper	1	25				
	2	25				
	Mean	25				

REPORT:

16. DETERMINATION OF EFFECT OF CONCENTRATION, SURFACE AREA AND VISCOSITY ON RATE OF EVAPORATION

AIM: To determine the effect of concentration, surface area and viscosity on rate of evaporation.

REQUIREMENTS: Beaker, Water-bath, Measuring cylinder, Glycerin, Purified water.

PRINCIPLE: - Evaporation is a process of vaporizing large quantities of volatile liquid to get a concentrated product.

The rate of evaporation depends on several factors such as temperature, viscosity, concentration of the slurry, vapour pressure, surface area, time of evaporation, films and deposits. Higher the concentration of dissolved solids (like sodium chloride), the lower the rate of evaporation. Higher the viscosity of the slurry, the lower the rate of evaporation. This is verified by taking slurries of different viscosities and subjecting to evaporation at constant temperature and surface area. The greater the surface area of the liquid. The greater will be the evaporation. For this reason, evaporation is conducted in evaporators with large heating surface area. It is verified by taking beakers of different surface area, i.e 50ml, 100ml and 250ml capacity. The rate of evaporation is calculated using the following formula:

$$\begin{aligned} \text{Rate of filtration} &= \frac{\text{Quantity of water evaporated (w)}}{\text{Time of heating (min)}} \\ &= \text{g/min} \end{aligned}$$

PROCEDURE:-

EFFECT OF CONCENTRATION

Procedure:

1. 2, 4, 6, and 8% w/v solutions of NaCl are prepared by dissolving 1, 2, 3, and 4 g of NaCl in 50 ml of water in beakers.
2. The beaker containing sodium chloride solutions are weighed (W_1 g). Weights are recorded in table.
3. All the beakers are heated in a water bath at constant temp. (70°C) for 30 mins.
4. All the heaters are weighed again after heating (W_2 g).
5. The difference between the weights is determined. The diff. reflects the amount of water evaporated during 30 minutes.

6. Rate of evaporation is calculated using the formula.
7. A graph is plotted by taking conc. on x-axis and rate of evaporation on y-axis.

OBSERVATIONS AND CALCULATIONS:

S. No.	Conc. of Sodium chloride solution, %w/v	Initial wt. of beaker + solution, W ₁ , g	Final wt. of beaker + solution, W ₂ , g	Wt. of water evaporated, w g	Time of heating, min	Rate of evaporation g/min.
				(3)-(4)		(5)/(6)
(1)	(2)	(3)	(4)	(5)	(6)	(7)
1.	2 %					
2.	4 %					
3.	6 %					
4.	8 %					

EFFECT OF SURFACE AREA

PROCEDURE:

1. Beakers measuring 50, 100 and 250 ml are cleaned.
2. 25 ml quantity of water is taken in each beaker.
3. Beakers containing water are weighed (initial wt. of beaker, w₁ g).
4. All the beakers containing water are heated in a water bath at constant temp. (70°C) for 30 mins.
5. After heating all the beakers are weighed again (final wt. of beaker, w₂ g).
6. The difference between the weights is determined, w g. The difference reflects the amount of water evaporated during 30 mins.
7. Radius (half of the diameter) of the beaker is noted. Using the radius, surface area of beakers is calculated, using the formula given below :-

$$\text{Surface area of beaker} = \pi r^2$$
8. Rate of evaporation is calculated using the formula
9. A graph is plotted by taking surface area on x-axis and rate of evaporation on y-axis.

OBSERVATIONS AND CALCULATIONS:

S. No.	Surface area of beaker, cm ²	Initial weight of beaker, w ₁ g	Final weight of beaker, w ₂ g	Wt. of water evaporated, w g	Time of heating, min	Rate of evaporation g/min.
				(3)-(4)		(5)/(6)
(1)	(2)	(3)	(4)	(5)	(6)	(7)
1.						
2.						
3.						
4.						

EFFECT OF VISCOSITY

PROCEDURE:

1. Different conc. of glycerin and water mixture are prepared in diff. beakers as shown in following Table:

Glycerin	Water	Conc.
5 ml	45 ml	10 %
10 ml	40 ml	20 %
15 ml	35 ml	30 %
20 ml	30 ml	40 %

2. The beakers containing glycerin-water mixtures are weighed (W_1 g), viscosities of these mixtures at room temp. are given in table. Weights are recorded in table.
3. All the beakers are heated in a water bath at constant temp. (70°C) for 30 mins.
4. All the heaters are weighed again after heating (W_2 g).
5. The difference between the weights is determined. The diff. reflects the amount of water evaporated during 30 minutes.
6. Rate of evaporation is calculated using the formula
7. A graph is plotted by taking viscosity on x-axis and rate of evaporation on y-axis.

OBSERVATIONS AND CALCULATIONS:

S. No	Conc. of glycerin water mix.	Viscosity of glycerin water mix	Initial wt. of beaker, W ₁ , g	Final wt. of beaker, W ₂ , g	Wt. of water evaporated, w g	Time of heating, min	Rate of evaporation g/min.
					(4)-(5)		(6)/(7)
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
1.	10 %	1.2823					
2.	20 %	1.8765					
3.	30 %	2.4020					
4.	40 %	2.9829					

REPORT :-

17. EFFECT OF TIME ON THE RATE OF CRYSTALLIZATION

AIM:- To study the crystallization behavior of potassium nitrate.

REQUIREMENTS: potassium nitrate, water bath,

PRINCIPLE: Potassium nitrate crystals can be obtained using shock cooling technique. The solid is added to a solvent continuously until the solid is dissolved. Such a solution is called as saturated solution. The rate of dissolution process is enhanced by increasing the temperature and agitation. Then the undissolved solid also goes into solution, When some solid remained undissolved, then such a solution is called as supersaturated solutions. When the temperature of supersaturated solutions is decreased rapidly the solubility of solute decreases. As a result the dissolved solid gets crystals growth. The extent of crystallization depends on the time of contact in low temperature. The crystals are collected by filtration and weighed. Yield is expressed as per cent weight of crystals obtained. A graph is plotted taking time versus percent weight crystals.

PROCEDURE:

1. Seventy five grams of potassium nitrate is accurately weighed (W_1 g).
2. Hundred ml of water is transferred into 250 ml beaker.
3. Beaker containing water is placed in constant temp. water bath maintained at 50°C .
4. Potassium nitrate is added into the water little by little, the solution is stirred with glass rod to dissolve the solute.
5. This process is continued until saturated solution (with little excess crystals) is formed.
6. Weight of potassium nitrate remained is weighed (W_2 g). Difference in the weights W g ($W_1 - W_2$) gives weight of potassium nitrate added into 100 ml water.
7. From this, 10 ml quantities of saturated solution are transferred into 9 test tubes.
8. All the test tubes are placed in an ice bath at once. Temp. of the solution decreases suddenly due to shock cooling forming supersaturated solution (Rate of cooling can be maintained constant by keeping the test tubes either in constant temp. water bath maintained at 20°C or in refrigerator). Nucleation and crystal growth takes place.
9. After 10 mins. The solution of first test tube is filtered to collect crystals.

10. This is repeated after every 10 mins. Thereafter using the solution of other test tubes.
11. All the crystals collected on the filter paper separately are subjected to drying.
12. Weights of each sample of crystals are recorded in table.
13. A graph is plotted taking time on x-axis and % wt. on crystals on y-axis.

OBSERVATION AND CALCULATIONS:

Data for crystallization of potassium nitrate

S.No	Test tube No.	Time, mins	Weight of crystals formed, g (b)	% wt. of crystals b/a X 100
1.	1	10		
2.	2	20		
3.	3	30		
4.	4	40		
5.	5	50		
6.	6	60		
7.	7	70		
8.	8	80		
9.	9	90		

a = Weight of potassium nitrate present in 10 ml of water, g

REPORT: Percent crystals of pott. nitrate crystals formed in 90 min =

18. MIXING INDEX

AIM: To determine the mixing index for the blending of salicylic acid and lactose in a blender.

REQUIREMENTS: Cylindrical Blender, Colorimeter and cuvettes, pipette (10 ml, 5 ml), test tube (20 ml), salicylic acid, Ferric nitrate solution (4% w/v), volumetric flask (100 ml), lactose.

PRINCIPLE: Cylindrical blender is used for mixing. In this experiment, dry powders of salicylic acid and lactose are mixed. During rotation of mixer, powders get mixed with each other. Sufficient time is allowed for mixing to get uniform blend.

During mixing, after every 10 mins, samples are drawn from three different places randomly. Each sample is subjected to determine the amount of ingredients present. Salicylic acid and lactose are soluble in water. Hence, to every sample dissolved in water, ferric nitrate is added to develop pink colour. Quantity of salicylic acid present is estimated by measuring absorbance of the colour colorimetrically at 547 nm. By knowing the amount of salicylic acid, the amount of lactose can be calculated.

Mixing index can be calculated using the following formula:

$$\text{Rate of Drying} = \sqrt{\frac{\Sigma(y-\check{y})^2}{n(1-\check{y})\check{y}}}$$

Ms = Mixing index.

N = number of samples.

\check{y} = true average composition of component A in the mixture.

Y = Actual composition of component A in a single sample.

PROCEDURE:

1. One g of salicylic acid and 50 g of lactose are weighed.
2. These two powders are placed in a cylindrical blender.
3. The blender is allowed to rotate on its own axis for 15 mins at 25 rpm.
4. The sample (500 mg) are drawn from three diff. places of the blender and placed in three diff. conical flasks. Labelled them as 1A, 1B, and 1C.
5. The blender is again allowed to rotate for another 10 mind.

6. Again three samples are drawn in a similar way as mentioned in step 4. They are transferred into three diff. conical flask and labeled as 2A, 2B and 2C.
7. Repeat the steps 5 after 45 mins. The samples are labeled as 3A, 3B, and 3C.
8. The samples are dissolved in water with continuous shaking. Finally, the volume is made upto 100 ml in each case.
9. From the volumetric flasks, 10 ml solutions are transferred into 20 ml test tubes.
10. 5 ml of ferric nitrate solution (4% w/v) is added to the above test tubes. All the solutions turn to purple colour.
11. The absorbance o the above solutions are measured at 547 nm using a colorimeter or spectrophotometer and reported in the table.
12. The content of salicylic acid and mixing index is calculated.

OBSERVATIONS AND CALCULATIONS:

Sampling time	Sample number	Absorbance	Conc. Of salicylic acid	Conc. Of salicylic acid in the sample y, mg (4) X 100	(y- \bar{y})	(y- \bar{y}) ²
(1)	(2)	(4)	(6)	(7)	(8)	
At 15 mins	1A					
	1B					
	1C					
			Average, \bar{y} =		$\Sigma(y-\bar{y})^2=$	
At 30 mins	2A					
	2B					
	2C					
			Average, \bar{y} =		$\Sigma(y-\bar{y})^2=$	
At 45 mins	3A					
	3B					
	3C					
			Average, \bar{y} =		$\Sigma(y-\bar{y})^2=$	

REPORT:

1. Mixing index after 15 mins, $M_{15} =$
2. Mixing index after 30 mins, $M_{30} =$
3. Mixing index after 45 mins, $M_{45} =$

PHYSICAL PHARMACEUTICS – I
LAB MANUAL
B. PHARMACY
2nd Year, 3rd Semester

1. DETERMINATION OF SOLUBILITY OF DRUG AT ROOM TEMPERATURE

Aim: To determine the solubility of benzoic acid at different temperatures.

Requirements: benzoic acid, distilled water, 0.1N Sodium hydroxide, phenolphthalein indicator, and filter paper, Measuring cylinder, funnel, beaker, conical flask, 10 ml bulb pipette, rubber bulb, burette, burette stand.

Principle:

The amount of drug dissolved in solution at a particular temperature is called solubility. Example: The solubility of paracetamol is 1 g in 70 ml water at 20 °C. The solubility of a drug is determined by preparing a saturated solution of the drug. A saturated solution is prepared by shaking excess quantity of the drug with the solvent for a long time (48 hours). This system is filtered and the saturated solution is analyzed for drug content by titration or suitable analytic method. In this experiment solubility of benzoic acid is determined by using distilled water. The amount of benzoic acid dissolved in the solvent is analyzed by titrating with 0.1 N Sodium hydroxide solution using phenolphthalein as indicator. When a drug (benzoic acid) has poor solubility in water, then the solubility of benzoic acid is improved by rise of temperature.

Procedure:

1. Take 50 ml of distilled water into a 100 ml beaker. Add required quantity of benzoic acid and shake vigorously for 30 minutes. If the added benzoic acid has dissolved, add further some amount of benzoic acid and continue shaking to obtain a saturated solution.
2. Heat the benzoic acid on the water bath up to 85°C.
3. Allow the temperature to fall gradually to 80°C.
4. Filter the contents into a clean dry beaker.
5. Titrate 10 ml of the filtrate with 0.1 N sodium hydroxide solution using phenolphthalein as indicator.
6. Continue the procedure and obtain data of solubility at 70,60,50,40 and 30°C temperatures.
7. Draw a plot by taking solubility of benzoic acid on y-axis and temperature on x-axis.
8. Calculate the solubility of benzoic acid in water.

Observations and Calculations:

S.NO	Temperature (°C)	Volume of sodium hydroxide consumed (ml) (V ₁)	Normality of benzoic acid (N ₂)	Solubility of benzoic acid (gm/ml)
1.	80			
2.	70			
3.	60			
4.	50			
5.	40			
6.	30			

Equivalent weight of benzoic acid is 122 gm

Normality of sodium hydroxide (N₁) is 0.1N

Volume of sodium hydroxide consumed is (V₁)

Volume of benzoic acid (V₂) is 10 ml sample taken at different temperatures.

$$\text{Normality of benzoic acid (N}_2\text{)} = \frac{N_1 V_1}{V_2}$$

$$\text{Solubility of benzoic acid} = N_2 \times \frac{122}{10}$$

Solubility of the drugs is expressed in various units in Merk Index

Term	Parts of solvent required for 1 part of solute
Very soluble	Less than 1part
Freely soluble	1 to 10 parts
Soluble	10 to 30 parts
Sparingly soluble	30 to 100 parts
Slightly soluble	100to 1000 parts
Very slightly soluble	1000 to 10,000 parts
Practically insoluble	More than 10,000 parts

Report: The solubility of benzoic acid in water _____ gm/ml at 80°C. As the temperature increases the solubility of benzoic acid is increased.

2. DETERMINATION OF P^{Ka} VALUE BY HALF NEUTRALISATION/ HENDERSON HASSEL BALCH EQUATION.

Aim: To determine the P^{Ka} value of the weak acid (acetic acid) by Henderson Hassel Balch equation.

Requirements: Acetic acid, distilled water, 0.1N Sodium hydroxide, 0.1N Oxalic acid, Measuring cylinder, funnel, beaker, conical flask, pH meter.

Principle:

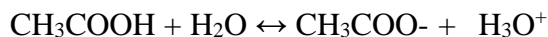
pH is a measure of hydrogen ion concentration, a measure of the acidity or alkalinity of a solution. The pH scale usually ranges from 0 to 14. Aqueous solutions at 25°C with a pH less than seven are acidic, while those with a pH greater than seven are basic or alkaline. A pH level of 7.0 at 25°C is defined as 'neutral' because the concentration of H₃O⁺ equals the concentration of OH⁻ in pure water. pH is given by equation as

$$\text{pH} = -\log[\text{H}^+]$$

where log is the base-10 logarithm and [H⁺] stands for the hydrogen ion concentration in units of moles per liter solution. pH can be measured by using Henderson Hassel Balch equation which is given by

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

To derive the dissociation constant (pK_a). Consider that the weak acid under gone partial dissociation.



$$\text{At equilibrium } K = \frac{K_1}{K_2} = \frac{(\text{CH}_3\text{COO}^-) (\text{H}_3\text{O}^+)}{(\text{CH}_3\text{COOH}) (\text{H}_2\text{O})}$$

where K₁ and K₂ are the rate constants of forward and backward reactions respectively. (H₂O) is a constant at about 55.3 moles/liter.

$$K_a = K \times 55.3 = \frac{(\text{CH}_3\text{COO}^-) (\text{H}_3\text{O}^+)}{(\text{CH}_3\text{COOH})}$$

$$\text{where } K_a \text{ is the dissociation constant. } (\text{H}_3\text{O}^+) = \frac{K_a (\text{CH}_3\text{COOH})}{(\text{CH}_3\text{COO}^-)}$$

Take $-\log$ on both sides, it becomes as

$$-\log (\text{H}_3 \text{O}^+) = -\log K_a -\log (\text{CH}_3\text{COOH}) + \log (\text{CH}_3\text{COO}^-)$$

$$\text{pH} = \text{p}K_a + \log \frac{(\text{CH}_3\text{COO}^-)}{(\text{CH}_3\text{COOH})}$$

$$\text{pH} = \text{p}K_a + \log \frac{(\text{salt})}{(\text{acid})}$$

In this study pH, $\text{p}K_a$ and K_a will be determined for acetic acid.

Procedure:

Prepare the buffer solutions using standard buffer tablets 4, 7 and 9.4. Calibrate the pH meter by using the buffer solutions. Take 0.05 ml of acetic acid in volumetric flask having capacity of 100 ml and make up the volume. This solution was taken in beaker and measures the pH of the solution. Finally calculate $\text{p}K_a$ of acetic acid by using the equation.

Observations and Calculations:

Molecular weight of acetic acid = 60.05

Weight per ml of solution = 1.0495

$$\text{pH} = -\log[\text{H}^+]$$

$$K_a = \frac{(\text{CH}_3\text{COO}^-) (\text{H}^+)}{(\text{CH}_3\text{COOH})} = \frac{(\text{H}^+)^2}{(\text{CH}_3\text{COOH})}$$

60.05 gm of acetic acid in 1000 ml = 1M

6.005 gm of acetic acid in 100 ml = 1M

$$(\text{CH}_3\text{COOH}) = \frac{\text{Volume of acetic acid} \times \text{weight per ml}}{\text{Weight of acetic acid for 1M}} = \frac{0.05 \times 1.0495}{6.005} = 0.0087 \text{ moles per liter.}$$

$$K_a = \frac{(10^{-\text{pH}})^2}{(\text{CH}_3\text{COOH})}$$

Therefore $\text{p}K_a = \text{pH} = -\log [\text{H}^+]$

Report:

The pH of the acetic acid solution is -----

The dissociation constant (K_a) of acetic acid is -----

The pK_a of the acetic acid solution is-----

3. DETERMINATION OF PARTITION COEFFICIENT OF BENZOIC ACID BETWEEN BENZENE AND WATER

Aim: To determine the partition coefficient of benzoic acid between benzene and water.

Requirements: Benzene, benzoic acid, 0.1N sodium hydroxide solution, phenolphthalein indicator, separating funnel, tripod stand, reagent bottles, two small beakers, measuring cylinder, conical flask, burette, burette stand, tile and digital balance.

Principle:

When a substance is added to a system containing two immiscible liquids, it distributes between the two liquids in a definite ratio." This is called Nernst distribution law. The added substance should have solubility in the two liquids for distribution to occur. This is known as the partition coefficient **K** of a substance between two liquids is given by the formula

$$\mathbf{K} = \frac{\text{Concentration of substance in organic layer}}{\text{Concentration of substance in aqueous layer}} = \frac{C_1}{C_2}$$

In the present experiment, distribution of benzoic acid between benzene and water is studied. Benzoic acid is an organic substance and has high solubility in benzene. It has less solubility in water. As a result, benzoic acid will partition preferably into benzene layer. The formula used for calculating partition coefficient of benzoic acid between benzene and water is given below. C_1 and C_2 are concentration of benzoic acid in organic and aqueous layer. In the present experiment, benzoic acid is shaken with benzene and water for 30 minutes to achieve distribution. Shaking is required to achieve distribution equilibrium. At equilibrium the speed of forward process is equal to the speed of backward process.

Benzoic acid is distributed as associated molecules in benzene layer and un associated molecules in aqueous layer. Hence the equation is given as follows.

$$\mathbf{K} = \frac{\sqrt{\text{Concentration of substance in organic layer}}}{\text{Concentration of substance in aqueous layer}} = \frac{\sqrt{C_1}}{C_2}$$

The partition coefficient **K** will be remains constant only if there is neither association nor dissociation of solute molecules in both the phases.

Procedure:

Preparation of 0.1N Sodium hydroxide: 4 gm of sodium hydroxide was dissolved in 1000 ml of distilled water and make up the final volume in volumetric flask.

1. Weigh the samples (250 mg, 500 mg and 750 mg) of benzoic acid into three reagent bottles and add 50 ml of benzene and 50 ml of water to all the three reagent bottles.
2. Keep the bottles on constant temperature water bath and Shake the bottles for 30 minutes.
3. Transfer the contents into a separating funnel and allow them to separate as two layers.
4. Collect the aqueous layer and titrate 10ml of sample with 0.1N sodium hydroxide solution using phenolphthalein as indicator.
5. Similarly collect the organic layer (benzene) and titrate 10ml of sample with 0.1N sodium hydroxide solution using phenolphthalein as indicator.
6. Calculate the partition coefficient of benzoic acid between benzene and water.

Observations and Calculations:

Equivalent factor: Each ml of 0.1N sodium hydroxide = 0.0122 gm of benzoic acid

Concentration of benzoic acid = Volume of sodium hydroxide consumed x 0.0122

S.NO	Volume of aqueous / benzene layer taken	Volume of sodium hydroxide consumed in ml	Concentration of benzoic acid	$\sqrt{C_1}$	Partition coefficient = $\frac{\sqrt{C_1}}{C_2}$
1	10 ml organic		C1=		
2	10 ml organic		C1 =		
3	10 ml aqueous		C2 =		
4	10 ml aqueous		C2 =		

Report: The partition coefficient of benzoic acid between benzene and water is _____.

4. DETERMINATION OF PARTITION COEFFICIENT OF IODINE BETWEEN CARBON TETRA CHLORIDE AND WATER

Aim: To determine the partition coefficient of iodine between carbon tetra chloride and distilled water.

Requirements: Iodine, carbon tetra chloride, 0.1N sodium thiosulphate solution, 0.005N sodium thiosulphate solution, starch mucilage as indicator, separating funnel, tripod stand, reagent bottles, two small beakers, measuring cylinder, conical flask, burette, burette stand, tile and digital balance.

Principle:

When a substance is added to a system containing two immiscible liquids, it distributes between the two liquids in a definite ratio.” This is called Nernst distribution law. The added substance should have solubility in the two liquids for distribution to occur. This is known as the partition coefficient **K** of a substance between two liquids is given by the formula

$$K = \frac{\text{Concentration of substance in organic layer } C_1}{\text{Concentration of substance in aqueous layer } C_2}$$

Where K is known as partition coefficient or distribution coefficient, C_1 and C_2 are the total concentrations of the solute in the two layers of organic and aqueous phases.

Procedure:

Preparation of saturated solution of Iodine: Dissolve the sufficient amount of iodine in carbon tetra chloride until some solid remains undissolved.

Preparation of 0.1N sodium thiosulphate solution: 26 gm of sodium thiosulphate and 0.2 gm of sodium carbonate was dissolved in 1000 ml of distilled water and make up the final volume in volumetric flask.

Preparation of 0.005N sodium thiosulphate solution: 1.3 gm of sodium thiosulphate and 0.01 gm of sodium carbonate was dissolved in 1000 ml of distilled water and make up the final volume in volumetric flask.

1. By means of a graduated pipette place about 30ml and 15ml of a saturated solution of iodine in carbon tetra chloride was prepared (stock solution) and properly labeled for glass stoppered bottles.
2. To these bottles add 100 ml of distilled water and shake the bottles for 20 minutes while keeping in water bath at room temperature. Keep it aside and allow them to separate as two phases of solution.
3. Withdraw 10 ml of the organic layer from first bottle carefully and titrate against 0.1 N sodium thiosulphate using starch solution as indicator.
4. Withdraw 10 ml of the organic layer from second bottle carefully and titrate against 0.1 N sodium thiosulphate using starch solution as indicator.
5. Similarly withdraw 10 ml of the aqueous layer from first bottle carefully and titrate against 0.005 N sodium thiosulphate using starch solution as indicator.
6. Similarly withdraw 10 ml of the aqueous layer from second bottle carefully and titrate against 0.005 N sodium thiosulphate using starch solution as indicator.
7. Calculate the partition coefficient of Iodine between carbon tetra chloride and water.

Observations and Calculations:

Titration of organic layer

S. No	Container	Volume of 0.1N sodium thiosulphate consumed in ml V ₁	Concentration of iodine in organic layer N ₂ = N ₁ V ₁ / V ₂
1	Bottle 1		
2	Bottle 2		

N₁= Normality of the sodium thiosulfate = 0.1N

V₁= volume of the sodium thiosulfate consumed =?

V₂= Volume of the organic layer = 10 ml

N₂= Normality (concentration) of the iodine =?

Titration of aqueous layer

S. No	Container	Volume of 0.005N sodium thiosulphate consumed in ml V1	Concentration of iodine in aqueous layer $N_2 = N_1 V_1 / V_2$
1	Bottle 1		
2	Bottle 2		

N_1 = Normality of the sodium thiosulphate = 0.005N

V_1 = volume of the sodium thiosulphate consumed = ?

V_2 = Volume of the aqueous layer = 10 ml

N_2 = Normality (concentration) of the iodine = ?

For bottle 1: $K = \frac{\text{Concentration of substance in organic layer}}{\text{Concentration of substance in aqueous layer}} = \frac{C_1}{C_2}$

For bottle 2: $K = \frac{\text{Concentration of substance in organic layer}}{\text{Concentration of substance in aqueous layer}} = \frac{C_1}{C_2}$

Report: The partition coefficient of iodine between carbon tetra chloride and distilled water was found to be -----.

5. DETERMINATION OF % COMPOSITION OF SODIUM CHLORIDE IN SOLUTION USING PHENOL WATER SYSTEM BY CST METHOD

Aim: To determine the % composition of sodium chloride in a solution using phenol water system by CST method.

Requirements: Phenol, distilled water, sodium chloride, thermometer, pipette, beaker, water bath and funnel.

Principle:

The temperature at which complete miscibility is reached as the temperature is raised or in some cases lowered used of two liquids that are partially miscible under ordinary conditions called also consolute temperature. The lower critical solution temperature (CST) or lower consolute temperature is the critical temperature below which the components of a mixture are miscible for all compositions. The word lower indicates that the LCST is a lower bound to a temperature interval of partial miscibility, immiscibility for certain compositions only. For example, the system triethylamine water has an LCST of 19°C , but not at higher temperatures. The Upper critical solution temperature or upper consolute temperature is the critical temperature above which the components of a mixture are miscible in all proportions. The word upper indicates that the UCST is an upper bound to a temperature range of partial miscibility, or miscibility for certain compositions only. For example, hexane nitrobenzene mixtures have a UCST of 19°C , so that these two substances are miscible in all proportions above 19°C but not at lower temperatures. When water and phenol are mixed together two layers are formed. The upper layer is solution of phenol in water. At a given temperature, composition of each solution is fixed and both solutions are in equilibrium. The two solution of different composition are existing in equilibrium with one another are known as conjugate solution. As the temperature increases, mutual solubility increases at a particular temperature this conjugate solution becomes completely miscible with one another. A temperature of which two conjugate solution are mutually soluble is called miscibility temperature. The miscibility temperature can be identifying as the disappearance of turbidity and reappearance of turbidity.

Procedure:

1. Prepare 50 ml of 1% w/v of sodium chloride in water and this stock solution is used for the preparation of different concentrations such as 0.1, 0.2, 0.4, 0.6, 0.8 and 1% v/v in the experiment.
2. Take 10 ml of stock solution each in boiling tubes and add 2 ml of phenol to each sample of stock solution.
3. Heat the mixture on water bath and note the temperature at which mixture becomes one layer in all the tubes (turbidity disappears). Note this miscibility temperature as $T_1^{\circ}\text{C}$.
4. Stirrer and thermometer are introduced in the sample tube. Continuously stir and observe the reappearance of turbidity of the mixture after cooling. Note this temperature at which turbidity reappears as $T_2^{\circ}\text{C}$.
5. Take the average of the temperature values that gives the CST of the solution. Similarly take 10 ml of the given unknown sample and add 2 ml of phenol to the sample and determine the CST of the sample.
6. Draw mutual solubility curve by plotting average miscibility temperature on Y-axis and percent composition of sodium chloride on X-axis. It will give the straight line. Using the graph read the percentage composition of unknown sample.

Observations and Calculations:

0.5 gm of sodium chloride in 50 ml gives 1% sodium chloride solution.

S. No	Sodium chloride solution [ml]	Distilled water [ml]	Percentage composition of sodium chloride	Turbidity disappears temperature [T1]	Turbidity reappears temperature [T2]	Average of temperature
1	1	9	0.1			
2	2	8	0.2			
3	4	6	0.4			
4	6	4	0.6			
5	8	2	0.8			
6	10	0	1.0			
7	unknown	Up to 10	unknown			

Report: The CST of unknown sample was found to be -----⁰C and the percent composition of sodium chloride in a solution (from graph) is ----- %.

6. DETERMINATION OF PARTICLE SIZE AND SIZE DISTRIBUTION USING SIEVING METHOD

Aim: To determine the average particle size and size distribution using sieving method.

Requirements: Granular sample, series of sieves (No: 20, 40, 60, 80, 100 and 120), mechanical sieve shaker.

Principle:

A sieve, or sifter, is a device for separating wanted elements from unwanted material or for characterizing the particle size distribution of a sample, typically using a woven screen such as a perforated mesh or metal. The particles sufficiently small will pass through and those that are over size retained on the sieve. A sieve will classify the particles as less than dimension of mesh (under size) and more than the dimension of mesh (over size) A Sieving method (or gradation test) is a procedure used to assess the particle size distribution (also called *gradation*) of a granular material by allowing the material to pass through a series of sieves of progressively smaller mesh size and weighing the amount of material that is stopped by each sieve as a fraction of the whole mass. In this experiment powder sample is passed through a set of sieves arranged with descending aperture size (coarsest sieve at the top) the weight remained on each sieve quantifies the particle size.

Procedure:

1. Arrange the sieves on the sieve shaker as larger aperture size on the top followed by smaller aperture size at the bottom.
2. Weigh accurately 100 g of the supplied powder, then place on the top sieve of the stack of sieves, cover and shake (mechanically) for 20 minutes.
3. Weigh the remaining powder on each sieve.
4. Enter the data of results in the table
5. Plot a graph between mean size of aperture on x-axis and percent weight retained on y-axis that gives the size distribution of particles.

Observations and Calculations:

S. No	Sieve number Passed and retained	Mean of aperture size (μm) d	Weight retained on sieve (gm) (frequency) n	% weight retained	Cumulative percent retained	weight size (n x d)
1	10/ 20					
2	20/40					
3	40/60					
4	60/80					
5	80/100					
6	100/120					
			$\Sigma n =$			$\Sigma n d =$

The average diameter of the particles is given by $D = \frac{\Sigma n d}{\Sigma n} =$

Sieve no (IP)	Aperture size (μm)
10	1700
20	840
30	500
40	420
50	300
60	250
70	210
80	180
100	150
120	125

Report: The average diameter of the particles of the sample----- μm

7. DETERMINATION OF PARTICLE SIZE AND SIZE DISTRIBUTION USING MICROSCOPY METHOD

Aim: To determine the particle size distribution of globule in emulsion by microscopy method.

Requirements: Microscope, glass slide, cover slips, Talc, starch, liquid paraffin

Principle:

The size of globules in an emulsion can be measured by microscopy method using an eyepiece micrometer. Eye piece micrometer has a small scale on it. The scale has to be calibrated using a stage micrometer. Stage micrometer is a glass slide having a scale on it. The scale is **1 mm** in length, and is divided into 100 parts.

The smallest division on the stage micrometer is 0.01 mm or **10 μm** in length. The exact value of each division on the eyepiece micrometer varies with every optical combination. Hence it should be calibrated with the stage micrometer for every optical combination.

If we want to measure the globule size under 45 x magnification, calibration of eyepiece micrometer should be done at 45 x magnification. If globule size is measured under 10 x magnification, calibration is to be done under 10 x. In the present experiment the calibration of eyepiece micrometer is done under 45 x magnification.

Emulsion is a heterogeneous system containing two immiscible liquids one dispersed in another. An ideal emulsion should have small globules, which are almost uniform in size. The smaller the globule size, the better is the stability of the emulsion. One of the methods for evaluating the stability of emulsions is globule size determination by microscopy technique.

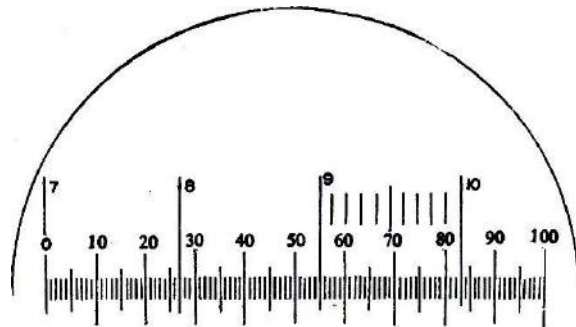
A calibrated eyepiece micrometer is used to measure the globule size in an emulsion. Microscopy method can be used to measure globules in the size range of 0.2 to 100 μm . In the present experiment, Agarose emulsion is suitably diluted with distilled water and the size of 100 globules is measured under 45 x magnification.

Average globule size and standard deviation of the globule size are calculated. A low standard deviation in globule size indicates that the globules are uniform in size.

Procedure:

Calibration of eyepiece micrometer

1. Replace the eyepiece of the microscope with the eyepiece micrometer.
2. Place the stage micrometer on the stage of the microscope and focus the scale under 10 x using coarse adjustment and fine adjustment knobs.
3. Make necessary adjustments so that the two scales are superimposed over one another.
4. Now rotate the nose piece of the microscope and focus the scale under 45 x magnification using fine adjustment knob only. Make necessary adjustments so that the two scales are parallel and superimposed over each other. Adjust the light condenser so that two scales are visible clearly.
5. Search for points of coincidence where the eyepiece division and stage division coincide perfectly. Let them be X and Y.
6. Find the number of divisions between X and Y on the stage and eyepiece micrometer.
7. From the distance between X and Y on stage micrometer and number of divisions between X and Y on eyepiece micrometer find the length of each division on the eyepiece micrometer.
8. The below figure shows the eyepiece micrometer and stage micrometer under 45 x magnification. The shorter lines of the numbered scale are the lines of the eye piece micrometer and the large lines are the lines of the stage micrometer.



9. In the above figure the 55th division of eyepiece micrometer coincides with 9th division of the stage micrometer (point X). Another point of coincidence is the 83rd division of the eyepiece micrometer and 10th division of stage micrometer (point Y). The calculations in calibration are given in the below table.

1.	Point X on eye piece micrometer	55
2.	Point Y on eye piece micrometer	83
3.	No. of eyepiece micrometer divisions between Y and X	$83 - 55 = 28$
4.	No. of stage micrometer divisions between X and Y	10
5.	Distance between X and Y on stage micrometer	$10 \times 10 \mu\text{m} = 100 \mu\text{m}$
6.	28 eye piece divisions is equal to	$100 \mu\text{m}$
7.	1 eyepiece division is equal to	$100/28 = 3.57 \mu\text{m}$

So, each eyepiece division on the eyepiece micrometer is equivalent to $3.57 \mu\text{m}$ under 45 X magnification.

Measurement of globule size:

1. Dilute Agarose emulsion suitably with distilled water. Spread one or two drops of the diluted emulsion on the glass slide and place a cover slip.
2. Focus the globules under 10x magnification using coarse adjustment and fine adjustment knobs.
3. Now rotate the nose piece of the microscope and focus the globules under 45 X magnification, using fine adjustment knob only. Make adjustments in the light condenser so that the globules and eyepiece micrometer divisions are clearly visible.
4. Measure the size of 100 globules by counting the number of eyepiece divisions occupied by each globule.
5. Calculate the average globule size and standard deviation associated with globule size.

Observations and Calculations:

Each eyepiece division on the eyepiece micrometer is equivalent to $3.57 \mu\text{m}$ under 45 X magnification.

Table with 100 particles

S. NO	No. of globules on each eye piece	Globule count (Tally marks)	Frequency (n)
1	1		
2	2		
3	3		
4	4		
5	5		

Frequency distribution table:

S. No	Size range (µm)	Mid-Point (µm) d	Frequency n	n x d
1	1-5			
2	5-10			
3	10-15			
4	15-20			
5	20-25			
			Σ n =	Σ n d =

The projected diameter of the globules of the sample is given by formula as

$$D_p = \frac{\sum n d}{\sum n}$$

Report: The projected diameter of the globules of the sample----- µm

8. DETERMINATION OF BULK DENSITY, TRUE DENSITY AND POROSITY

Aim: To determine the Bulk Density, True Density and Porosity for the given sample of powders.

Requirements: Measuring cylinder, Bulk density apparatus, specific gravity bottle, powder sample (magnesium oxide or lactose or talc)

Principle:

The bulk density denotes the total density of the material as it exists. The bulk volume includes the true volume, volume of inter particle spaces and intra particle pores. The packing is mainly responsible for bulk.

Bulk density is defined as:

$$\text{Bulk density, } \rho_b = \frac{\text{weight of the powder}}{\text{bulk volume of the powder}}$$

Since bulk volume includes the true volume, volume of inter particle spaces (voids) and intra particle pores, determining the volume of the powder using a measuring cylinder may be appropriate.

The true density is the density of the powder i.e., material exclusive of voids (inter particle spaces) and intra particle pores. The density is dependent on the type of atoms in a molecule, arrangement of the atoms in a molecule and the arrangement of molecules in a sample. The most common methods used in the determination of the true density are gas (helium or nitrogen) displacement and liquid displacement (mercury, organic liquid) methods. Helium and nitrogen gases obey ideal gas law at ambient temperatures and pressures. Helium penetrates the smallest pores and crevices. Therefore, helium densitometry gives a value closer to its true density. True density is given by the equation as

$$\text{True density, } \rho_t = \frac{\text{weight of the powder}}{\text{mass of displaced liquid}} \times \text{density of liquid}$$

Porosity can provide the information about the nature of powder sample whether the sample of powder is porous or nonporous. Porosity can be expressed in terms of densities as given below

$$\text{Porosity} = \frac{\text{True density} - \text{Bulk density}}{\text{True density}}$$

Procedure: For bulk density

1. Pass the required quantity of powder through a sieve no: 20. Weigh the 10 gm of powder and place in 100 ml capacity of measuring cylinder.
2. Fix the measuring cylinder to the bulk density apparatus and note the volume of the powder.
3. Finally determine the bulk density from the formula.

$$\text{Bulk density, } \rho_b = \frac{\text{weight of the powder}}{\text{bulk volume of the powder}}$$

Procedure: For True density

1. Take a clean, dry specific gravity bottle and weigh the empty specific gravity bottle as W1. Fill the bottle with water and keep the stopper and weigh the specific gravity bottle with water as W2. Remove water and wash the bottle with acetone.
2. Dry the bottle with the help of hot-air dryer and fill it with powder weigh it as W3. Now fill the powder in to the bottle and pour liquid to displace the voids in the sample of powder in the bottle and weigh as W4. Use the formula to determine the true density.

$$\text{True density, } \rho_t = \frac{\text{weight of the powder}}{\text{mass of displaced liquid}} \times \text{density of liquid} = \frac{(W3-W1)}{(W2-W1) - (W4-W3)} \times \text{density of liquid}$$

Procedure: For Porosity

Note down the values of true density and bulk density and substitute in the given equation.

$$\text{Porosity} = \frac{\text{True density} - \text{bulk density}}{\text{True density}}$$

Observations and calculations:

Weight the empty specific gravity bottle = W1

Weight the specific gravity bottle + water = W2

Weight of specific gravity bottle + powder = W3

Weight of specific gravity bottle + powder + water = W4

Density of water at room temperature = 0.99 g /cc.

Report: Bulk density of the powder is -----g/cc. True density of the powder is ----- g/cc.

Porosity of the powder is-----.

9. DETERMINE THE ANGLE OF REPOSE AND INFLUENCE OF LUBRICANT ON ANGLE OF REPOSE

Aim: To determine the angle of repose of given powder/granules and influence of lubricant on angle of repose.

Requirements: Powder sample (lactose), funnel, burette stand, talc, magnesium stearate.

Principle:

Angle of repose is defined as the maximum angle possible between the surface of the pile of powder and horizontal plane. The angle of repose is designated by θ and given by equation.

$$\tan \theta = h/r$$

Where h =height of the pile r =radius of the pile

The lower the angle of repose the better the flow properties. When granules are placed in the hopper and allowed to slide down onto the die for compression, it forms a pile. The angle of repose may be calculated by measuring the height of the pile and the radius of the base with ruler. During the flow through the hopper, the granules exhibit internal flow and demixing (i.e. the tendency of the powder to separate into layers of different sizes). Flow of granules is hindered on account of frictional forces. Lubricants are those substances which promote the flow of the granules or powder material by reducing the friction between the particles.

Relationship between the angle of repose and powder flow

Angle of repose	Powder flow
< 25	Excellent
25-30	Good
30-40	Passable
> 40	Very poor

Procedure:

1. Select a glass funnel, which has a round stem of 15 to 30 mm diameter with a flat edge. Fix the funnel with a clamp to the iron stand.
2. Place a 100 gm of granules into funnel while blocking the orifice of the funnel by thumb.

3. Remove the thumb and the granules flow down onto the graph paper and form a cone shaped pile.
4. Adjust the funnel clamp so that the gap between the bottom of the funnel stem and peak of the powder pile is about 3mm.
5. Repeat the steps until appropriate gap is maintained.
6. Finally pour the granules back into funnel and allow to flow. Measure the height of the pile using two rulers. Keep one ruler vertically and another horizontally to touch the peak of the pile. Then read the value on the vertical scale.
7. Record the reading this value represents height and also measure diameter and radius. Substitute the values in equation to obtain the angle of repose.
8. Repeat this procedure for two trials and take an average.
9. Add the lubricant in low concentration (1.0 g of talc or 0.2g of magnesium stearate) to the granules and mix them thoroughly.
10. Add further increments of lubricant and determine the angle of repose.
11. Repeat this procedure with further additions of 1.0 g of talc until optimum concentration is obtained

Observations and calculations:

Angle of repose without lubricant

Trials	Height [h] cm	Diameter[d] cm	Radius[r] cm	h/r	$\theta = \text{Tan}^{-1} \text{h/r} [^\circ]$
I					
II					

$$\text{Angle of repose} = \text{Tan } \theta = \text{h/r} = \theta = \text{Tan}^{-1} \text{h/r}$$

Angle of repose with lubricant (Talc)

(Talc) gm	Height [h] cm		Diameter[d] cm		Radius[r] cm		h/r	
	Trail I	Trail II	Trail I	Trail II	Trail I	Trail II	Trail I	Trail II
0								
1								
2								
3								
4								
5								
6								

$$\text{Angle of repose} = \tan^{-1} \theta = \tan^{-1} \frac{h}{r} = \theta = \tan^{-1} \frac{h}{r}$$

Report:

The angle of repose of given powder/granules is -----

When lubricant is added, the angle of repose of the material is -----.

As the concentration of the lubricant is increased the angle of repose of the material is increased

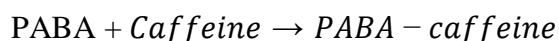
10. DETERMINATION OF STABILITY CONSTANT AND DONOR ACCEPTOR RATIO OF PABA – CAFFEINE COMPLEX BY SOLUBILITY METHOD

Aim: To determine the complex stability constant and donor acceptor ratio of caffeine and para-amino benzoic acid (PABA) by solubility method.

Requirements: Volumetric flask, beakers, conical flasks, pipette, burette, funnel, Para amino benzoic acid, sodium hydroxide (0.025N), caffeine, phenolphthalein indicator, Whatman filter paper.

Principle:

Complex compounds are defined as those molecules in which most of the bonding structures can be described by classical theories of valency between atoms or molecules. Complexes possess some properties, which are different from those of its components. Properties such as solubility, light absorption, conductance, partitioning behavior and chemical reactivity are studied to confirm the formation of complexes. For example, para Amino Benzoic acid and caffeine form complexes in solution. This results in enhanced solubility of PABA at low concentrations of caffeine. Further increase in concentration of caffeine results in decreased solubility of PABA. Therefore, the change in the solubility profile is taken as a criterion to decide the complexation behavior. The equation for the formation of complex is



The interaction may be due to dipole-dipole force or hydrogen bonding between the polar carbonyl groups of caffeine and hydrogen atom of the acid. The secondary interaction may probably occur between the non-polar parts of the molecules. The analysis of complexes generally involves the estimation of two parameters. These are represented by equations as

1. Stoichiometric ratio = $\frac{[\text{caffeine in complex}]}{[\text{PABA in complex}]}$
2. Complex stability constant = $\frac{[\text{PABA-caffeine}]}{[\text{PABA}][\text{ caffeine}]}$

In this method, caffeine is taken in different concentrations in a series of flasks. Excess quantity of PABA (same quantity) is added to all the flasks. These flasks are corked and agitated at a

constant temperature bath, until equilibrium is attained. The samples are filtered and saturated solution is collected and analyzed for drug content. The corresponding concentrations are substituted in equations 1 and 2.

Procedure:

Caffeine stock solution (0.1N): Weigh 1.949 gm of anhydrous caffeine and transfer into 100 ml of volumetric flask and add distilled water to make up final volume.

Para amino benzoic acid (PABA): Weigh accurately the required number of samples containing 200 mg of Para amino benzoic acid.

Preparation of 0.025 N sodium hydroxide: 1 gm of sodium hydroxide was dissolved in 1000 ml of distilled water and make up the final volume in volumetric flask.

1. Prepare various concentrations of caffeine (use 100ml conical flasks or beaker). The concentrations of caffeine are given in table. Transfer the samples of PABA into each flask containing the above caffeine solutions. Fix the flasks in a constant temperature bath and shake them for 30 minutes to attain equilibrium.
2. Filter the above solutions with Whatman filter paper and 10 ml of filtrate was taken and titrated with 0.025N sodium hydroxide using phenolphthalein indicator. Complete the titration of all samples and process the data in the table.
3. Draw a plot between concentration of caffeine on X-axis and concentration of PABA on Y-axis. Calculate the complex stability constant and donor acceptor ratio of caffeine and para amino benzoic acid (PABA) using the equations.

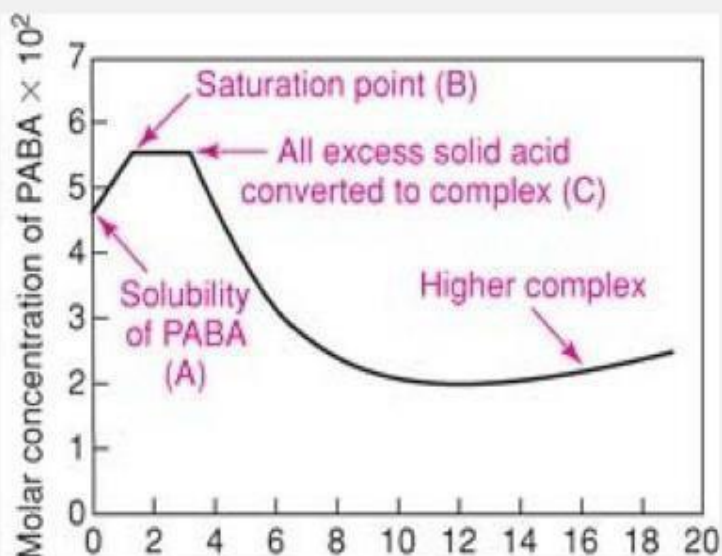
Observations and calculations:

Concentration of caffeine solution

S. No	Caffeine solution (ml)	Distilled water (ml)	Concentration of caffeine mol/liter
1	0	20	0
2	2	18	1
3	4	16	2
4	6	14	3
5	8	12	4
6	10	10	5
7	12	8	6
8	16	4	8

Analysis of complex:

S. No	Concentration of caffeine mol/liter	Volume of sodium hydroxide consumed in ml	Concentration of PABA mol/liter
1	0		
2	1		
3	2		
4	3		
5	4		
6	5		
7	6		
8	8		



The solubility of *para*-aminobenzoic acid (PABA) in the presence of caffeine.

Report: The complex stability constant of caffeine and para amino benzoic acid (PABA) is----
 The donor acceptor ratio of caffeine and para amino benzoic acid (PABA) is-----.

11. DETERMINATION OF STABILITY CONSTANT AND DONOR ACCEPTOR RATIO OF COPPER-GLYCINE COMPLEX BY pH TITRATION METHOD

Aim: To determine the complex stability constant ($\log \beta$) and donor acceptor ratio (n) of Copper - Glycine complex pH by titration method.

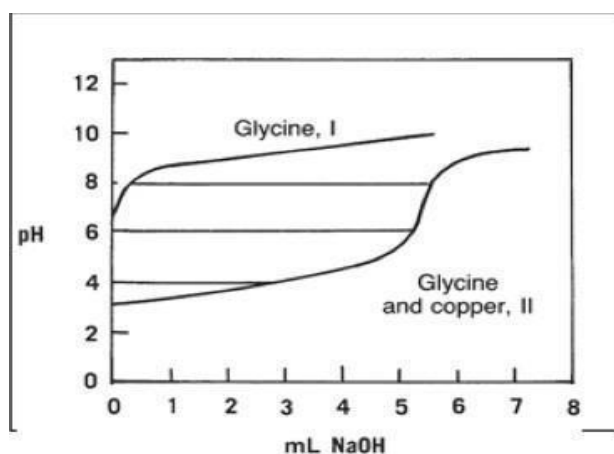
Requirements: Volumetric flask, beakers, conical flasks, pipette, burette, funnel, cupric chloride Glycine, sodium hydroxide (0.25N), phenolphthalein indicator, pH meter, buffer tablets, pH 7, 4 and 9.4.

Principle:

Complexation of copper ions with Glycine can be represented by the following equation.



Because of two protons are formed in the reaction of equation the addition of glycine to a solution containing cupric ions should result in a decrease in pH. Titration curves can be obtained by adding a strong base to a solution of glycine and to another solution containing glycine and a copper salt and plotting the pH against the equivalents of base added. The results of such a potentiometric titration are shown in the figure. The curve for the metal-glycine mixture is well below that for the glycine alone, and the decrease in pH shows that complexation is occurring throughout most of the neutralization range.



Titration of glycine and cupric glycine complex solution. The difference in pH for a given quantity of base (sodium hydroxide) added indicates the occurrence of a complex.

The average number of ligand groups bound per metal ion can be given by equation as

$$n = \frac{\text{Total concentration of ligands bound}}{\text{Total concentration of metal ion}}$$

The horizontal distance represents the amount of alkali added in the titration. This quantity is equal to the concentration of ligand bound to metal at any pH. The total concentration of metal ion taken initially is known. Thus, n can be calculated. The stability constant (β) and pH of free glycine are related as

$$p(A) = \frac{1}{2} \log \beta \text{ at } n = 1$$

$p(A)$ can be estimated using the equation

$$p(A) = pK_a - pH - \log([HA]_{\text{initial}} - [NaOH])$$

Where pK_a is dissociation constant of glycine, (9.69)

[NaOH] is concentration of sodium hydroxide in mol/lit.

Procedure:

Preparation of 0.25N sodium hydroxide: 10 gm of sodium hydroxide was dissolved in 1000 ml of distilled water and make up the final volume in volumetric flask.

Preparation of Glycine solution (3.34×10^{-2} mol/lit): Weigh 250 mg of glycine and transfer into a 100 ml of volumetric flask, add distilled water and make up the volume.

Complex solution (Glycine – 3.34×10^{-2} mol/lit; cupric chloride – 9.45×10^{-3} mol/lit): Weigh 250 mg of glycine and 160 mg of cupric chloride and transfer into a 100 ml of volumetric flask, add distilled water and make up the volume. Prepare two such samples.

Kinetic method:

1. Transfer the 75 ml of glycine solution into a beaker. Measure the pH of the solution. Gradually add the 0.25N of sodium hydroxide solution to the glycine solution.
2. Transfer the 75 ml of glycine-cupric complex solution into a beaker. Measure the pH of the solution. Gradually add the 0.25N of sodium hydroxide solution to the glycine-cupric complex solution.
3. Identify the range where the sudden increase in pH is obtained in the complex solution.
4. Take another sample of 75 ml of complex solution and add 1 ml increment up to 5 ml to the complex mixture and report the data.

5. Titrate the complex solution further (note: If sudden increase in pH is observed between 5 to 6 ml. Then in the final analysis, increments of 0.2 ml of sodium hydroxide should be added, i.e., 5.0, 5.2, 5.4, 5.6, 5.8 and 6.0).
6. After 6.0 ml, add 1 ml increments to the complex mixture and report the data.
7. Draw a graph between volume of sodium hydroxide added on x-axis and pH on the y-axis by using data obtained in the titration of glycine and complex solution.

Observations and calculations:

Data for analysis of complex of cupric-glycine by pH titration method

Glycine solution		Preliminary study Complex solution		Final readings Complex solution	
Volume of sodium hydroxide solution (ml)	pH	Volume of sodium hydroxide solution (ml)	pH	Volume of sodium hydroxide solution (ml)	pH
0		0		0	
1		1		1	
2		2		2	
3		3		3	
4		4		4	
5		5		5	
6		6		5.2	
		7		5.4	
		8		5.6	
				5.8	
				6.0	
				7.0	
				8.0	

Report: The complex stability constant ($\log \beta$) of Copper - Glycine complex pH by titration method is----- . The donor acceptor ratio (n) of Copper - Glycine complex pH by titration method is ----- .

INDUSTRIAL PHARMACY LAB MANUAL

B. PHARM

3rd YEAR, 5th SEMESTER

INDUSTRIAL PHARMACY LAB

List of Experiments:

Sl. NO.	List of Experiments
1	Preformulation study for prepared granules
2	Preparation and evaluation of Paracetamol tablets
3	Preparation and evaluation of Aspirin tablets
4	Coating of tablets
5	Preparation and evaluation of Tetracycline capsules
6	Preparation of Calcium Gluconate injection
7	Preparation of Ascorbic Acid injection
8	Preparation of Paracetamol Syrup
9	Preparation of Eye drops
10	Preparation of Pellets by extrusion spheronization technique
11	Preparation of Creams (cold / vanishing cream)
12	Evaluation of Glass containers (As per IP)

EXPERIMENT NO: 01

PREFORMULATION STUDY FOR PREPARED GRANULES

AIM: To perform different Preformulation studies of prepared granules.

REQUIREMENTS: Measuring cylinder, Funnel, Sieves, Mortar & pestle, Spatula.

PRINCIPLE: Preformulation is defined as the phase of research and development in which preformulation studies characterize physical and chemical properties of a drug molecule in order to develop safe, effective and stable dosage form. The Objective of preformulation study is to develop the elegant, stable, effective and safe dosage form by establishing kinetic rate profile, compatibility with the other ingredients and establish Physico-chemical parameter of new drug substances. The major preformulation studies/parameters of granules are as follows:

- 1. Bulk density:** It is defined as ratio of total mass of the powder to the bulk volume of powder. It gives an idea about tablet porosity and its relationship with disintegration time and hardness of a tablet. It is measured by pouring weighed powder into a measuring cylinder and the volume is noted down. It is expressed in gm/ml and is given by

$$D_b = M/V_o$$

Where,

M= Mass of powder,

V_o = Bulk volume of powder

- 2. Tapped density:** It is defined as ratio of total mass of the powder to the tapped volume of powder. Tapped volume is measured by tapping the powder to constant volume. It is expressed in gm/ml and is given by:

$$D_t = M/V_t$$

Where,

M= Mass of powder,

V_t = Tapped volume of powder

- 3. Angle of repose (Θ):** It is the maximum angle possible between surface of pile of powder and the horizontal plane, can be used to measure frictional forces in a powder.

$$\Theta = \tan^{-1}(h/r)$$

Where,

Θ= angle of repose

H height of the powder in cm, R is the radius of heap of powder

Relationship between Angle of repose and flow property

Angle of repose(θ)	Type of flow
<25	Excellent
25-30	Good
30-40	Passable
>40	Very poor

- 4. Carr's Compressibility Index:** It indicates the ease with which a material can be induced to flow; it is expressed as a percentage and is given by

$$I = (D_t - D_b) / D_t \times 100$$

Where,

D is the tapped density of the powder.

D_b is the bulk density of the powder.

Relationship between Carr's index and flow property

Carr's index	Type of flow
5-15	Excellent
12-15	Good
15-22	Fair
23-30	Poor
33-38	Very poor
>40	Extremely poor

- 5. Hausner's ratio:** It indicates the flow properties of the powder and is measured by the ratio of tapped density to the bulk density.

$$\text{Hausner's ratio} = (\text{Tapped density}) / (\text{Bulk density}) \times 100$$

Values of Hausner's ratio : < 1.25: good flow and > 1.25: poor flow

If Hausner's ratio is between 1.25-1.5, flow property can be improved by addition of glidants.

- 6. Size and Size Distribution Analysis:** The particle-size distribution (PSD) of a powder, or granular material, is a list of values or a mathematical function that defines the relative amount, (typically by mass) of particles present according to size.

The size and shape distribution of the metal particles impacts powder behavior during die filling, compaction, and sintering, and therefore influences the physical properties of the parts created. In the pharmaceutical industry the size of active ingredients influences critical characteristics including content uniformity, dissolution and absorption rates.

Measurement Techniques:

1. Sieve Analysis
2. Air elutriation analysis
3. Photo analysis
4. Optical counting methods
5. Electro resistance counting methods
6. Sedimentation techniques
7. Laser diffraction methods

The way PSD is usually defined by the method by which it is determined. The most easily understood method of determination is sieve analysis, where powder is separated on sieves of different sizes. Thus, the PSD is defined in terms of discrete size ranges: e.g. "% of sample between 45 μm and 53 μm ", when sieves of these sizes are used. The PSD is usually determined over a list of size ranges that covers nearly all the sizes present in the sample. However, the idea of the notional "sieve", that "retains" particles above a certain size, and "passes" particles below that size, is universally used in presenting PSD data of all kinds.

The PSD may be expressed as a "range" analysis, in which the amount in each size range is listed in order. It may also be presented in "cumulative" form, in which the total of all sizes "retained" or "passed" by a single notional "sieve" is given for a range of sizes. Range analysis is suitable when a particular ideal mid-range particle size is being sought, while cumulative analysis is used where the amount of "under-size" or "over-size" must be controlled.

PROCEDURE:

Bulk density and tapped density: Pass a quantity of sample sufficient to complete the test through a sieve, if necessary, to break up agglomerates. Into a measuring cylinder of 100 ml, gently introduce, without compacting, approximately 15g of the test sample and weighed. Carefully level the powder without compacting, if necessary, and read the unsettled apparent

Volume to the nearest graduated unit. Calculate the bulk density by applying the above formula. The tapped volume is obtained by mechanically tapping the measuring cylinder containing the sample of 15 gm with a fixed drop of 14 ± 2 mm at a nominal rate of 300 drops per mins until a constant volume is observed. Then calculate the tapped density by using the above formula.

After getting the value of bulk density and tapped density, **Carr's Compressibility Index and Hausner's ratio** is calculated by using the formula.

Angle of repose: The static angle of repose was measured according to the fixed funnel and free standing cone method. A funnel was clamped with its tip 2cm above a graph paper placed on a flat horizontal surface. The powders were carefully poured through the funnel. Block the orifice of the funnel by thumb. Fill the powder in the funnel and remove the thumb immediately. After emptying the powder from the funnel, measure the height of the pile and diameter.

Size and Size Distribution Analysis: Arrange all the sieves on the shaker one above the other in increasing opening order i.e. decreasing sieve number, the one with powder sample occupying the upper most position. Weigh about 50g (W) of given sample and place it over the top sieve (Lowest sieve number). Shake the sieve either mechanically or electrically for a period of half an hour.

The powder retained on each sieve is collected and weighed separately. The percentage weight retained on each sieve is calculated by,

Percentage powder retained=

$$\frac{\text{weight of powder that have retained over the sieve}}{\text{weight of total powder taken for experiment}} \times 100$$

OBSERVATIONS:

S. No	Sieve number passed or retained	Arithmetic mean size of opening(μm)	Average size of the particle	Weight retained on a sieve(gm)	%weight retained	Cumulative percentage of oversized particles	Cumulative percentage of undersized
1	10/16	1350		W_1			
2	16/22	855		W_2			
3	22/40	517.5		W_3			
4	40/60	287.5		W_4			
5	60/85	142.5		W_5			
6	85/100	27.5		W_6			

REPORT:

The preformulation parameters of the prepared granules were found to be:

Bulk density:

Tapped density:

Carr's Compressibility Index:

Hausner's ratio:

Angle of repose:

Size distribution analysis: The given sample is size separated by the sieves.

Their frequency distribution curve of the particle was plotted.

The average particle size---- μm were found to be maximum of ---%

The average particle size----- μm were found to be minimum of ---- %

The cumulative size distribution curve were also plotted and the total average particle size is found to be-- μm

INTRODUCTION TO TABLETS

Tablets may be defined as the solid unit dosage forms containing one or more medicaments and excipients, prepared either by molding or compression. It comprises a mixture of active substances and excipients in powder or granule form. The excipients include diluents, binders or granulating agents, glidants and lubricants to ensure efficient tablet compression, disintegrants to promote tablet break-up in the digestive tract, sweeteners or flavors to enhance taste and pigments to make tablets visually attractive.

ADVANTAGES:

1. Tablets offer the greatest compatibilities of all oral dosage forms for the greatest dose precision and the least content variability.
2. Their cost is lowest of all oral dosage form.
3. They are lightest and compact.
4. Easiest and cheapest to package and ship.
5. They have better physical and chemical stability and exert physiological activity of drug.
6. Special forms to facilitate patient compliance eg: - sustained release, extended release formulations.
7. Suitable for large scale economical production.

DISADVANTAGES:

1. Unsuitable for infants and children and patients who cannot swallow.
2. Delayed onset of action compared to liquid orals and parenterals.
3. Drugs with poor wetting, slow dissolution properties, optimum absorption high in GIT or combination of above features make tablet manufacturing difficult.
4. Bitter tasting drugs, drugs with objectionable odor or drugs that are sensitive to oxygen or atmospheric moisture may require encapsulation or entrapment prior to compression.

DIFFERENT TYPES OF TABLETS

They are generally divided as

- A. Compressed tablets
- B. Moulded tablets/ Tablets triturates.

CLASSIFICATION OF TABLETS ACCORDING TO USAGE:

(A) Tablets ingested orally:

1. Compressed tablet, e.g. Paracetamol tablet

2. Multiple compressed tablet

- a. Layered tablets
- b. Press coated/Dry coated Tablets

3. Repeat action tablet

4. Delayed release tablet, e.g. Enteric coated Bisacodyl tablet

5. Sugar coated tablet, e.g. Multivitamin tablet

6. Film coated tablet, e.g. Metronidazole tablet

7. Chewable tablet, e.g. Antacid tablet

(B) Tablets used in oral cavity:

1. Buccal tablet, e.g. Vitamin-C tablet
2. Sublingual tablet, e.g. Nitroglycerin tablet
3. Troches or lozenges
4. Dental cone

(C) Tablets used to prepare solution:

1. Effervescent tablet, e.g. Dispirin tablet (Aspirin)
2. Dispensing tablet, e.g. Enzyme tablet (Digiplex)
3. Hypodermic tablet
4. Tablet triturates e.g. Enzyme tablet

(D) Tablets administered by other Routes

1. Implantation tablets
2. Vaginal tablets

FORMULATION OF TABLETS:

In addition to active ingredient, tablet contains a number of inert materials known as additives or excipients.

Different excipients are:

1. Diluents
2. Binders and adhesives
3. Disintegrants
4. Lubricants and glidants
5. Colouring agents
6. Flavoring agents

7. Sweetening agents

1. Diluents (Fillers)

Diluents are used to make required bulk of the tablet when the drug dosage is inadequate to produce the bulk. Secondary reason is to provide better tablet properties such as improve cohesion, to permit use of direct compression manufacturing or to promote flow.

- a. Diluents for wet granulation
 - i. Lactose (hydrous): Most widely used. Lactose reacts with certain amine drugs / proteins in the presence of metal stearates (lubricants) resulting in the tablet discoloration with time. Such a reaction is known as *Millard reaction*(*Browning reaction*)
 - ii. Anhydrous lactose
 - iii. Dicalcium phosphate and calcium sulfate: Excellent for water sensitive drugs because they contain appreciable water content and have low affinity to atmospheric moisture.
 - iv. Bentonite and kaolin
- b. Diluents for dry granulation and direct compression
 - i. Spray dried lactose
 - ii. Directly compressible starches (corn, wheat or potato). They act as lubricant, binder and disintegrants
 - iii. Colloidal silica
 - iv. Sodium chloride used for dental cones
 - v. Mannitol, sorbitol, sucrose, dextrose (These agents can also be used as binder in solution form or for wet granulation)

2. Binders and Adhesives: These materials are added to hold powders together to form granules to promote cohesive compacts for directly compressed tablet.

Example: Acacia, tragacanth- Solution for 10-25% Conc. Cellulose derivatives- Methyl cellulose, Hydroxy propyl methyl cellulose, Polyvinylpyrrolidone (PVP)- 2% conc. Starch paste- 5-15% solution.

3. Disintegrants: Added to a tablet formulation to facilitate its breaking or disintegration when it comes in contact with water in GIT. Disintegrants acts by three mechanisms

- a. Swelling e.g., alginates, starch, PVP ect.

- b. Improving penetration of aqueous liquids (wetting agents) e.g., SLS, clays
- c. Liberation of gas from effervescent base, e.g., NaHCO₃ and citric acid.

Superdisintegrants: Swells up to ten fold within 30 seconds when contact water.

Example: Crosscarmellose- cross-linked cellulose, Crosspovidone- cross-linked povidone (polymer), Sodium starch glycolate- cross-linked starch.

4. Lubricants: These are added for the following reasons

- Prevents adhesion of the tablet material to the surface of dies and punches.
- Reduce inter-particle friction; improve the rate of flow of tablet granulation.
- Facilitate ejection of the tablets from the die cavity.

Example: Lubricants- Stearic acid, Stearic acid salt – Stearic acid, Magnesium stearate, Talc, PEG (Polyethylene glycols). Glidants- Corn Starch – 5-10% conc, Talc-5% conc., Silica derivative – Colloidal silicas such as Cab-O-Sil, Syloid, Aerosil in 0.25-3% conc.

Glidants are intended to promote flow of the tablet granulation or powder materials by reducing the friction between the particles.

5. Coloring agent: The use of colors and dyes in a tablet has three purposes:

- (i) It makes the tablet more esthetic in appearance.
- (ii) Colour helps the manufacturer to identify the product during its preparation.

All colorants used in pharmaceuticals *must be approved and certified by the FDA (Food & Drug Administration)*. Dyes are generally listed as FD&C (Food, Drug & Cosmetic Dyes) dyes and D&C (Drug & Cosmetic Dyes).

Example: FD & C yellow 6-sunset yellow FD & C yellow 5- Tartrazine FD & C green 3- Fast Green FD & C blue 1- Brilliant Blue FD & C blue 2 – Indigo carmine D & C red 3- Erythrosine. D & C red 22 – Eosin Y

6. Flavoring agents: Flavors are usually limited to chewable tablets or other tablets intended to dissolve in the mouth. Flavor oils are added to tablet granulations in solvents, are dispersed on clays and other adsorbents or are emulsified in aqueous granulating agents (i.e. binder). Usually, the maximum amount of oil that can be incorporated to a granulation without influencing its tableting characteristics is 0.5 to 0.75% w/v.

6. Sweetening agents: The use of sweeteners is primarily limited to chewable tablets.

e.g - Sugar.

Mannitol-72% as sweet as sugar, cooling & mouth filling effect

Saccharin- Artificial sweetener, 500 times sweeter than sucrose. *Disadvantages*: it has a bitter after taste and carcinogenic

Aspartame (Searle) - widely replacing saccharin. *Disadvantage* – lack of stability in presence of moisture

MANUFACTURING METHODS OF TABLETS: In the tablet-pressing process, it is important that all ingredients be dry, powdered, and of uniform grain size as much as possible. The main guideline in manufacture is to ensure that the appropriate amount of active ingredient is equal in each tablet so ingredients should be well-mixed. Compressed tablets are exerted to great pressure in order to compact the material. If a sufficiently homogenous mix of the components cannot be obtained with simple mixing, the ingredients must be granulated prior to compression to assure an even distribution of the active compound in the final tablet. Two basic techniques are used to prepare powders for granulation into a tablet: wet granulation and dry granulation.

Powders that can be mixed well do not require granulation and can be compressed into tablets through Direct Compression.

The manufacturing of tablet dosage form is basically done by two methods, such as

- 1) Wet Granulation (most products)
- 2) Direct Compression

WET GRANULATION: Wet Granulation is a process of size enlargement whereby small particles are gathered into larger permanent aggregates in which the original particles can still be identified. Granulation usually refers to processes whereby agglomerates with sizes ranging from to 2.0 mm are produced. The most important reasons for a granulation step prior to tableting are to:

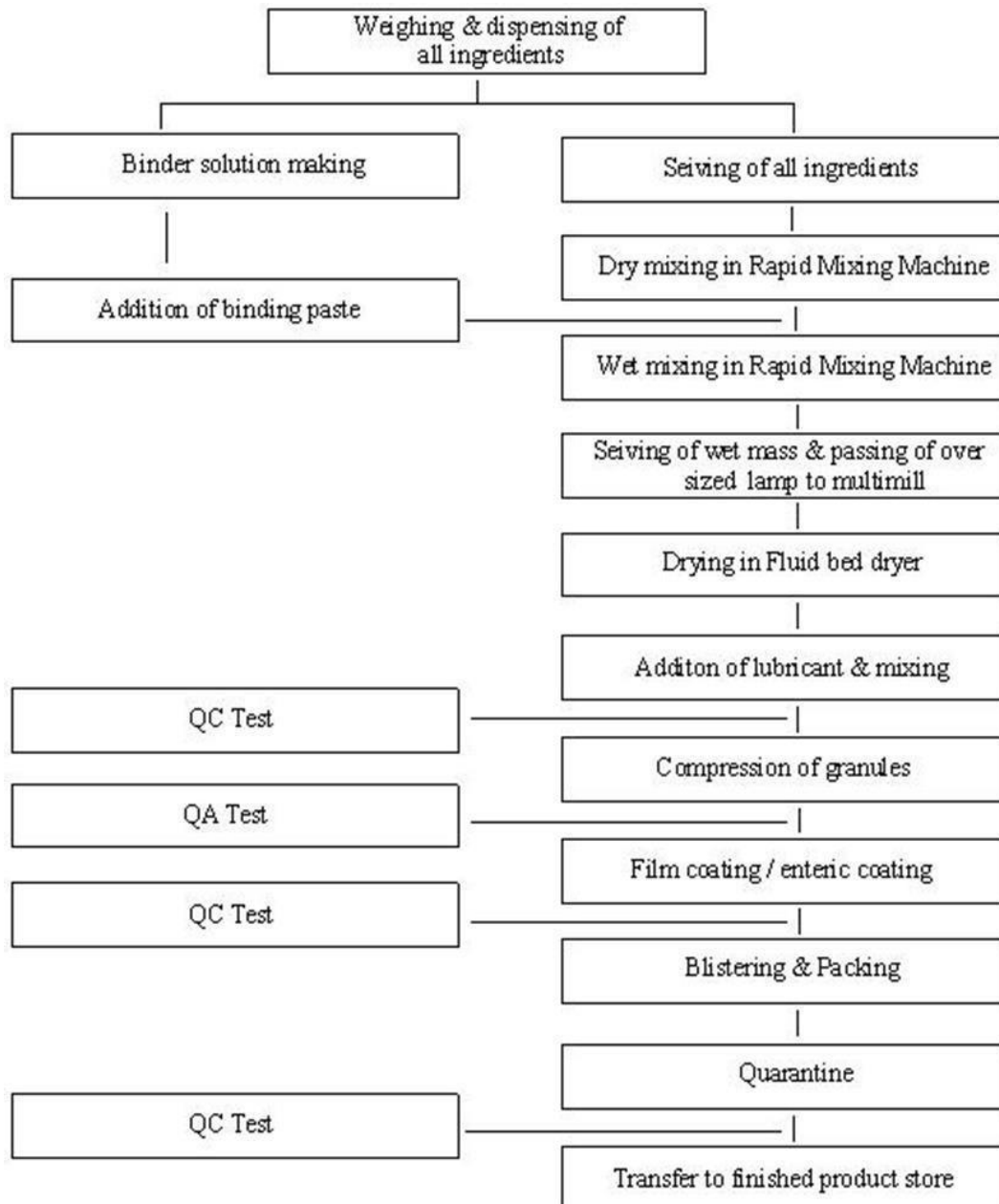
- Improve the flow properties of the mix and hence the uniformity of the dose.
- Prevent segregation of the ingredients.
- Improve the compression characteristics of the tablet mixture.
- Reduce dust during handling

The flow ability of the tablet mixture improves because the granules are larger and more spherical than the primary particles. Larger particles usually flow better than small particles (e.g. compare the flow ability of crystal sugar with powder sugar). In the hopper of tablet machines, small particles tend to segregate from the larger ones because of the vibration of the machine.

This causes higher concentrations of small particles at the bottom of the hopper. After granulation all particles are bound tight in the right amount in the granules, which prevents segregation of the small particles

Process Flow Chart

(Wet granulation method)



Equipment's used in wet granulation method:

1. Electronic Balance
2. Sieve
3. Rapid Mass Granulator (RMG)
4. Multimill
5. Fluid Bed Dryer
6. Double Cone Blender
7. Vat for the preparation of granulating fluid

DIRECT COMPRESSION: In the direct compression method, directly compressible filler (also called a filler-binder) is blended with the active(s), a lubricant and a disintegrating agent. Such free flowing directly compressible fillers make direct compression possible and practical. These include anhydrous lactose, unmilled dicalcium phosphate dihydrate, microcrystalline cellulose (e.g., Avicel PH 101), and modified (spray processed) lactose (e.g., Ludipress). Modified starch, e.g. Starch 1500 flows better and compresses better than original starch, but are not as effective as other materials as the sole filler-binder. Generally, Starch 1500 is used as a component of a direct compression filler system, most likely for its disintegrating property, i.e., as a more compactible and better flowing substitute for starch. Certain materials like mannitol, sorbitol and modified sucrose are particularly useful in formulating direct compression chewable tablets.

Direct compression method can be classified as

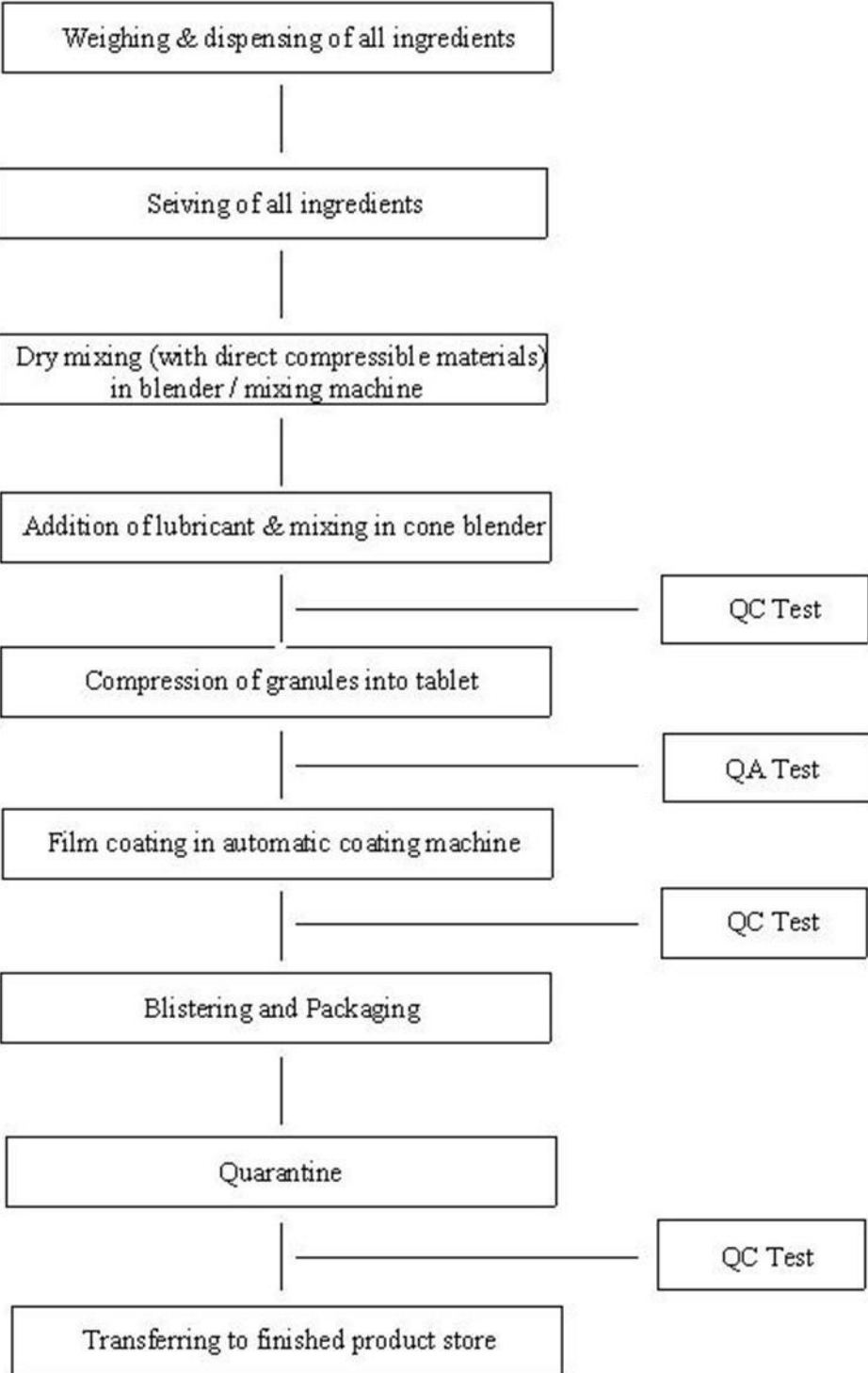
- a) Direct Compression with direct compressible materials and
- b) Direct Compression by Slugging method

Equipment's used in direct compression method:

1. Electronic Balance
2. Sieve
3. Double cone blender
4. Rotary Press

Process flow chart

(Direct Compression with direct compressible materials)



EXPERIMENT NO: 2

PREPARATION OF PARACETOMOL TABLETS

AIM: To prepare and submit 10 paracetamol (100 mg) tablets by wet granulation method.

REQUIREMENTS: Mortar and pestle, spatula, beaker, Sieve

PRINCIPLE: Tablet is an important solid dosage form which is usually prepared with the aid of suitable pharmaceutical excipients. Tablets may vary with size, shape, cut, hardness, thickness. Their disintegration and dissolution characteristics and other aspects change depending on their intended use and method of manufacturing.

Compressed tablets are mainly prepared by 3 basic methods

- Wet granulation
- Dry granulation
- Direct compression

Wet granulation is the widely used method for the production of compressed tablets. Steps involved in wet granulation method are

- a) Weighing and blending of ingredients
- b) Preparing a damp mass by adding wet binder
- c) Converting the damp mass into wet granules
- d) Drying of granules
- e) Sizing the granules by dry screening
- f) Addition of lubricants
- g) Formation of tablets by compression

During the preparation process each step may influence the quality of tablet produced. In this preparation paracetamol used as API (antipyretic), lactose as adjuvant, starch (purified) as binding agent, starch monohydrate as disintegrant, magnesium stearate as lubricant and talc as Glidant.

Ingredients table (Formula):

S. NO	INGREDIENTS	1 TABLET	10 TABLETS	PURPOSE
1	PARACETAMOL(API)			Analgesic & Antipyretic
2	STARCH (PURIFIED)			Binding agent
3	LACTOSE MONOHYDRATE			Diluent
4	STRARCH MONOHYDRATE			Disintegrant
5	TALC			Glidant
6	MG.STEARATE			Lubricant

PROCEDURE:

- a) **Preparation of starch mucilage:** Dissolve 5mg of starch in 100ml of distilled water then resulting mixture is heated on a water bath until the starch is gelatinized by the formation of mucilage.
- b) Divide disintegrating agent (starch monohydrate) into 2 portions to incorporate during wet granulation and after drying of granules to act as an intragranular and extra granular disintegrant.
- c) **Wet Granulation:** Accurately weigh and mix the specified amount of paracetamol and other excipients (except half of the disintegrating agent and lubricant) until uniform powder is formed by geometric mixing.
- d) A damp mass of the mixture is prepared by adding appropriate amount of the 5% starch mucilage and kneading by hand.
- e) Wet mass is subsequently passes through a 6/10 mesh sieve/screen to form wet granules. Resulted granules are spread evenly on a large piece of paper in a tray and dried at 40°C-60°C for 30min in an oven.
- f) Dried granules are passed through a sieve 16 or 20 # and mixed with remaining half of the disintegrating agent and lubricant.
- g) Resulting granules mixture is compressed in a tablet compression machine to obtain tablets.
- h) Prepared tablets are stored properly for further evaluation.

REPORT: Paracetamol tablets were prepared by wet granulation method and submitted.

EXPERIMENT NO: 03

EVALUATION OF PARACETOMOL TABLETS

AIM: To evaluate prepared paracetamol tablets.

REQUIREMENTS: Beaker, Test tubes, Test apparatuses

PRINCIPLE:

Evaluation parameters of tablets:

APPEARANCE:

Tablet from each formulation were randomly selected and organoleptic properties such as color, taste, and shape were evaluated.

HARDNESS TEST:

The tablet hardness is defined as the force required to break a tablet in a diametric direction. A tablet was placed between two anvils. Force was applied to anvils and crushing strength that causes the tablet to break was recorded. The hardness was measured using Monsanto hardness tester.

THICKNESS:

The thickness of tablets was determined using a Vernier calliper. Three tablets from each batch were used, and average values were calculated.

FRIABILITY TEST:

The friability of tablets was determined using Roche Friabilator. It is expressed in percentage (%). Ten or twenty tablets were initially weighed and revolved at 25 rpm for 4 min. The tablets were then reweighed after removal of fines and the percentage of weight loss was calculated. The % friability was then calculated by,

$$F = (W_{\text{initial}} - W_{\text{final}}) \times 100 / W_{\text{initial}}$$

Acceptance criteria for % friability % weight loss should be less than 1%.

WEIGHT VARIATION TEST:

Twenty tablets were selected randomly from each batch and weighed individually on electronic balance. The individual weighed is then compared with average weight for the weight variations. The following percentage deviation in weight variation is allowed (U.S.P).

Average weight	% difference
130 mg or less	10
130 – 324 mg	7.5
More than 324 mg	5

DISINTEGRATION TIME TESTING:

It was determine using USP tablet disintegration test apparatus, using 900 ml of distilled water without disk at room temperature. Test was performed on 6 tablets. One tablet each is kept in all six tubes. The tubes travel upward and downward in water at $37^{\circ}\text{C}\pm 2^{\circ}\text{C}$. The time taken for all the six tablets to break down and pass through the mesh at the bottom of the tube is noted. The tablets pass the test if all the six tablets disintegrate within the prescribed time (Less than 30 mins for uncoated tablets as per U.S.P).

IN VITRO DRUG RELEASE STUDY:

The release rate of paracetamol from tablets was determined using United States Pharmacopeia (USP) Dissolution Testing Apparatus Type-II. The dissolution test was performed using 900ml of 5.8pH phosphate buffer, at $37^{\circ}\text{C}\pm 0.5^{\circ}\text{C}$ and 50 rpm. A sample (10ml) of the solution was withdrawn from the dissolution apparatus hourly and the samples were replaced with fresh dissolution medium. The samples were filtered through a 0.45μ membrane filter. Absorbance of these solutions was measured at 243 nm using a Thermospectronic-1 UV/V double-beam spectrophotometer. Cumulative percentage drug release was calculated using an equation obtained from a standard curve.

REPORT: The evaluation tests are performed and all the tablets are found to be in the acceptable limits.

EXPERIMENT NO: 04

PREPARATION OF ASPIRIN TABLETS

AIM: To prepare and submit 10 Aspirin (100 mg) tablets by wet granulation method.

REQUIREMENTS: Mortar and pestle, spatula, beaker, Sieve

PRINCIPLE: Tablet is an important solid dosage form which is usually prepared with the aid of suitable pharmaceutical excipients. Tablets may vary with size, shape, cut, hardness, thickness. Their disintegration and dissolution characteristics and other aspects change depending on their intended use and method of manufacturing.

Compressed tablets are mainly prepared by 3 basic methods

- Wet granulation
- Dry granulation
- Direct compression

Wet granulation is the widely used method for the production of compressed tablets. Steps involved in wet granulation method are

- h) Weighing and blending of ingredients
- i) Preparing a damp mass by adding wet binder
- j) Converting the damp mass into wet granules
- k) Drying of granules
- l) Sizing the granules by dry screening
- m) Addition of lubricants
- n) Formation of tablets by compression

During the preparation process each step may influence the quality of tablet produced. In this preparation Aspirin used as API (Aspirin, also known as acetylsalicylic acid, is a medication used to treat pain, fever, or inflammation), lactose as adjuvant, acacia as binding agent, starch monohydrate as disintegrant, magnesium stearate as lubricant and talc as Glidant.

Ingredients table (Formula):

S.NO	INGREDIENTS	1 TABLET	10 TABLETS	PURPOSE
1	ASPIRIN (API)			Treat Pain, Fever, Or Inflammation
2	ACACIA			Binding agent
3	LACTOSE MONOHYDRATE			Diluent
4	STRARCH MONOHYDRATE			Disintegrant
5	TALC			Glidant
6	MG.STEARATE			Lubricant

PROCEDURE:

- a) Divide disintegrating agent (starch monohydrate) into 2 portions to incorporate during wet granulation and after drying of granules to act as an intragranular and extra granular disintegrant.
- b) **Wet Granulation:** Accurately weigh and mix the specified amount of Aspirin and other excipients (except half of the disintegrating agent and lubricant) until uniform powder is formed by geometric mixing.
- c) A damp mass of the mixture is prepared by adding appropriate amount of the acacia and drop wise addition of water.
- d) Wet mass is subsequently passes through a 6/10 mesh sieve/screen to form wet granules. Resulted granules are spread evenly on a large piece of paper in a tray and dried at 40°C-60°C for 30min in an oven.
- e) Dried granules are passed through a sieve 16 or 20 # and mixed with remaining half of the disintegrating agent and lubricant.
- f) Resulting granules mixture is compressed in a tablet compression machine to obtain tablets.
- g) Prepared tablets are stored properly for further evaluation.

REPORT: Aspirin tablets were prepared by wet granulation method and submitted.

EXPERIMENT NO: 05

EVALUATION OF ASPIRIN TABLETS

AIM: To evaluate prepared Aspirin tablets.

REQUIREMENTS: Beaker, Test tubes, Test apparatuses

Evaluation parameters of tablets:

APPEARANCE:

Tablet from each formulation were randomly selected and organoleptic properties such as color, taste, and shape were evaluated.

HARDNESS TEST:

The tablet hardness is defined as the force required to break a tablet in a diametric direction. A tablet was placed between two anvils. Force was applied to anvils and crushing strength that causes the tablet to break was recorded. The hardness was measured using Monsanto hardness tester.

THICKNESS:

The thickness of tablets was determined using a Vernier caliper. Three tablets from each batch were used, and average values were calculated.

FRIABILITY TEST:

The friability of tablets was determined using Roche Friabilator. It is express in percentage (%). Ten or twenty tablets were initially weighed and revolved at 25 rpm for 4 min. The tablets were then reweighed after removal of fines and the percentage of weight loss was calculated. The % friability was then calculated by,

$$F = (W_{\text{initial}} - W_{\text{final}}) \times 100 / W_{\text{initial}}$$

Acceptance criteria for % friability % weight loss should be less than 1%.

WEIGHT VARIATION TEST:

Twenty tablets were selected randomly from each batch and weighed individually on electronic balance. The individual weighed is then compared with average weight for the weight variations.

The following percentage deviation in weight variation is allowed (U.S.P).

Average weight	% difference
130 mg or less	10
130 – 324 mg	7.5
More than 324 mg	5

DISINTEGRATION TIME TESTING:

It was determine using USP tablet disintegration test apparatus, using 900 ml of distilled water without disk at room temperature. Test was performed on 6 tablets. One tablet each is kept in all six tubes. The tubes travel upward and downward in water at $37^{\circ}\text{C}\pm 2^{\circ}\text{C}$. The time taken for all the six tablets to break down and pass through the mesh at the bottom of the tube is noted. The tablets pass the test if all the six tablets disintegrate within the prescribed time (Less than 30 mins for uncoated tablets as per U.S.P).

IN VITRO DRUG RELEASE STUDY:

The release rate of Aspirin from tablets was determined using United States Pharmacopeia (USP) Dissolution Testing Apparatus Type-II. The dissolution test was performed using 900ml of 5.8pH phosphate buffer, at $37^{\circ}\text{C}\pm 0.5^{\circ}\text{C}$ and 50 rpm. A sample (10ml) of the solution was withdrawn from the dissolution apparatus hourly and the samples were replaced with fresh dissolution medium. The samples were filtered through a 0.45μ membrane filter. Absorbance of these solutions was measured at 265 nm using a Thermospectronic-1 UV/V double-beam spectrophotometer. Cumulative percentage drug release was calculated using an equation obtained from a standard curve.

REPORT: The evaluation tests are performed and all the tablets are found to be in the acceptable limits.

EXPERIMENT NO: 06

FORMULATION OF FILM COATED TABLETS OF PARACETAMOL

AIM: To prepare 10 tablets of paracetamol film coated tablets.

REQUIREMENTS: Mortar and pestle, Sieve, Beaker, Glass rod

PRINCIPLE: All drugs have their own characteristic, like some drugs are bitter in taste or have an unpleasant odor, some are sensitive to light or oxides, some are hygroscopic in nature. Because of this reasons, tablet coating is the choice of option to solve such problems in conventional dosage form. Tablet film coating is performed by two types, one is aqueous film coating (generally water is used as a solvent) and non-aqueous film coating (generally organic solvents are used). Some problems are associated with the non-aqueous film coating like safety of employees (as most of the solvents are dangerous, smell, and they are not good to breathe), atmospheric pollution etc. But key problem is with the approval of the regulatory authority. High quality aqueous film coating must be smooth, uniform and adhere satisfactorily to the tablet surface and ensure chemical stability of a drug. Coating may be applied to a wide range of oral solid dosage forms, including tablets, capsules, and multi particulate and drug crystals. When coating composition is applied to a batch of tablets in a coating pan, the tablet surfaces become covered with a tacky polymeric film. Before the tablet surface dries, the applied coating changes from a sticky liquid to tacky semisolid and eventually to a non-stick dry surface. The entire coating process is conducted in a series of mechanically operated acorn-shaped coating pans of galvanized iron stainless steel or copper. The smaller pans are used for experimental, developmental, and pilot plant operations, while the larger pans for industrial production.

Necessity of Tablet Coating:

- A number of reasons can be suggested, like: The core contains a material which has a bitter taste in the mouth or has an unpleasant odor. Coating will protect the drug from the surroundings with a view to improve its stability.
- Coating will increase the ease by which a tablet can be ingested by the patient.
- Coating will develop the mechanical integrity; means coated products are more resistant to mishandling (abrasion, attrition, etc.)
- The core contains a substance which is incompatible in the presence of light and subject to atmospheric oxidation, i.e. a coating is added to improve stability.

- The coated tablets are packed on high-speed packaging machine. Coating reduces friction and increases packaging rate.
- Coating can modify the drug release profile, e.g., enteric coating, osmotic pump, pulsatile delivery.

Ingredients table (Formula):

Name of the ingredient	Quantity (%w/w)
Cellulose acetate	6.3
PEG 400	0.7
Acetone	89
De-ionized water	4

PROCEDURE: Paracetamol uncoated tablets are prepared by wet granulation method. The prepared tablets are then coated with film coating solution prepared as below.

Film coating solution preparation: The coating solution was prepared by dissolving PEG in water followed by addition of this solution to acetone. Cellulose acetate was then added to the above mixture and stirred to achieve a clear solution.

The coating process was performed in a Vector Hi-Coater LDCS (batch size, 1.5 kg, with inclusion of placebo tablets) at a product temperature of 28°C. Coated tablets were dried in a vacuum drying oven at 40°C for 24 hours to remove residual solvent and moisture.

REPORT: 10 tablets of paracetamol film coated tablets are prepared and submitted.

EXPERIMENT NO: 07

FORMULATION OF ENTERIC COATED TABLETS OF OMEPRAZOLE

AIM: To prepare 10 tablets of enteric coated tablets of omeprazole.

REQUIREMENTS: Mortar and pestle, Beaker, Sieve, Glass rod

PRINCIPLE:

Enteric coatings are primarily used for the purpose of:

- Maintaining the stability of APIs that are unstable when exposed to the acidic conditions of the gastric milieu. Such API's include erythromycin, pancreatic, and the class of proton pump inhibitors, such as omeprazole.
- Minimizing the side effects (eg, nausea, and gastric irritation and bleeding) that can occur with APIs such aspirin and certain nonsteroidal inflammatory compounds.
- Creating opportunities for "night-time dosing" strategies, where the intent is to allow the dosage form to be consumed at bed-time, and permit effective blood levels of the API to be attained just prior to waking.
- Facilitating colonic drug delivery. The functionality of enteric coatings is, for the most part, mediated by a change in pH of the environment to which the enteric-coated product is exposed. Enteric polymers remain unionized (and thus, insoluble) at low pH values, and begin to dissolve at a pH value of approximately 5.0–5.5.

Ingredients table (Formula for uncoated tablet):

Name of the ingredient	Quantity(mg)
Dry mix	
Omeprazole	20
Lactose hydrate	92.86
Sodium starch glycolate	4
Sodium lauryl sulphate	1.5
Magnesium hydroxide	6
Binding solution	
Hydroxyl propyl cellulose and water	1
Lubrication	
Talc	1.32
Mg. stearate	1.32
Seal coating	
HPMC E15 & purified water	3
Enteric coating	
HPMC phthalate	7
Triacetin	0.394
Talc	0.394
Isopropyl alcohol	q.s
Acetone	q.s

PROCEDURE:

Wet granulation:

- Weigh accurately require quantity of Omeprazole, Lactose , Sodium starch glycolate , Sodium Lauryl Sulfate, Magnesium hydroxide and pass through sieve no #40
- Prepare a binding solution by dissolving HPC in water and stir binder solution for 10 minutes, this solution is added to dry mix to form granules.
- The prepared granules are dried and lubricated with lubricants.
- The lubricated granules are then compressed to form immediate release tablets.

Seal Coating: Prepare sub coating solution using HPMC or PVA in Purified water. Coat the uncoated tablets with the seal coating suspension to achieve required weight gain.

Enteric coating: Prepare enteric coating suspension using HPMC Phthalate, Triacetin, and Talc in Acetone-IPA mixture. Coat the uncoated tablets with the enteric coating suspension to achieve required weight gain.

REPORT: 10 tablets of omeprazole enteric coated tablets are prepared and submitted.

EXPERIMENT NO: 08

PREPARATION AND EVALUATION OF HARD GELATIN

CAPSULES OF TETRACYCLINE HYDROCHLORIDE

AIM: To prepare and evaluate hard gelatin capsules of tetracycline hydrochloride.

REQUIREMENTS: Mortar and pestle, beaker, test tubes, spatula, glass rod, Test apparatuses

PRINCIPLE: Hard gelatin capsule shells are used in most commercial medicated capsules. The community pharmacist also uses hard gelatin capsules in the extemporaneous compounding of prescriptions. The empty capsule shells are made of gelatin, sugar, and water. As such, they can be clear, colourless, and essentially tasteless; or they may be colored with various dyes and made opaque by adding agents such as titanium dioxide. Most commercially available medicated capsules contain combinations of colorants and opaquants to make them distinctive, many with caps and bodies of different colors. Gelatin is obtained by the partial hydrolysis of collagen obtained from the skin, white connective tissue, and bones of animals. In commerce, it is available in the form of a fine powder, a coarse powder, shreds, flakes, or sheets. Gelatin is soluble in hot water and in warm gastric fluid; a gelatin capsule rapidly dissolves and exposes its contents. Gelatin, being a protein, is digested by proteolytic enzymes and absorbed. Advantages of hard gelatin capsule are rapid drug release possible, flexibility of formulation and sealed HGCs are good barriers to atmospheric oxygen. Disadvantages of this dosage form are very bulky materials are a problem, filling equipment process is slower than tablets, generally more costly than tablets, but must judge on a case-by-case basis; concern over maintaining proper shell moisture content.

Tetracycline is used to treat a wide variety of infections, including acne. It is an antibiotic that works by stopping the growth of bacteria. This antibiotic treats only bacterial infections. It will not work for viral infections (e.g., common cold, flu). First Tetracycline hydrochloride granules are prepared by using wet granulation technique by using required ingredients. Then these granules are filled in the hard gelatin capsule shell

FORMULA:

Name of the ingredient	Quantity (mg)
Tetracycline hydrochloride	100
Microcrystalline cellulose	38
PVPK30	6
Magnesium stearate	4
Talc	2
Alcohol	q.s

PROCEDURE:**Formulation of Granules of Tetracycline hydrochloride:**

Tetracycline hydrochloride granules were prepared by wet granulation method. Specified quantity of tetracycline hydrochloride, micro crystalline cellulose and PVP K30 will be weighed and mixed uniformly. Required quantity of alcohol drop wise incorporated to the blend. Wet granules will be passed through sieve #10 & air dried for 15 minutes. The dried granules will then be passed through sieve #22. Required quantity of magnesium stearate & talc were added to the granules. The prepared granules were then added to the Size #3 empty hard gelatin capsule.

Evaluation of prepared capsule of tetracycline hydrochloride:

Weight Variation Test: Twenty capsules were selected randomly from each batch and weighed individually on electronic balance. The individual weighed is then compared with average weight for the weight variations. The % difference should be 10%.

Disintegration time Testing: It was determine using disintegration test apparatus, using 900 ml of distilled water with disk (in case capsule floats) at room temperature. Test was performed on 6 capsules. One capsule each is kept in all six tubes. The tubes travel upward and downward in water at $37^{\circ}\text{C}\pm 2^{\circ}\text{C}$. The capsules pass the test if no drug or particles other than capsule fragments remained on the mesh or tube. The time taken for that is considered as disintegration time.

In vitro drug release study: The release rate of Tetracycline hydrochloride from capsule was determined using United States Pharmacopeia (USP) Dissolution Testing Apparatus Type-II. The dissolution test was performed using 900ml of 5.8pH phosphate buffer, at $37^{\circ}\text{C}\pm 0.5^{\circ}\text{C}$ and 50 rpm. A sample (10ml) of the solution was withdrawn from the dissolution apparatus hourly

and the samples were replaced with fresh dissolution medium. The samples were filtered through a 0.45 μ membrane filter. Absorbance of these solutions was measured at 344 nm using a Thermospectronic-1 UV/V double-beam spectrophotometer. Cumulative percentage drug release was calculated using an equation obtained from a standard curve.

REPORT: Tetracycline hydrochloride hard gelatin capsules were prepared and evaluated.

EXPERIMENT NO: 09

PREPARATION OF CALCIUM GLUCONATE INJECTION

AIM: To prepare and submit 10 ml Calcium gluconate injection.

REQUIREMENTS: Beaker, Glass rod, Funnel, Filter paper, Ampoule

PRINCIPLE: Injections are sterile solutions, emulsions or suspensions. They are prepared by dissolving, emulsifying or suspending an active ingredient and any other substances in water for injection. Injecting is the act of giving medication by use of syringe and needle to obtain the desired therapeutic effect taking into account the patient's safety and comfort. It is suitable for those drugs that are altered or not absorbed by other methods of administration.

Calcium gluconate is a mineral supplement and medication. As a medication it is used by injection into a vein to treat low blood calcium, high blood potassium, and magnesium toxicity. Supplementation is generally only required when there is not enough calcium in the diet.

Calcium Gluconate is the calcium salt of gluconic acid, an oxidation product of glucose, and contains 9.3% calcium, which is about one-third of the calcium in strength of calcium chloride USP. Since it is soluble to the extent of only one part in 30 parts of cold water, the 10% solution is supersaturated and is stabilized by the addition of calcium saccharate tetrahydrate 0.46% w/v.

FORMULA:

Ingredients	1 ml injection	10 ml injection
calcium gluconate monohydrate	98 mg	
calcium saccharate tetrahydrate	4.6 mg	
Water for injection upto	1 ml	

PROCEDURE: calcium gluconate monohydrate and calcium saccharate tetrahydrate are dissolved in water for injection in a beaker and makes upto required volume. Filter it and take 1 ml of the filtrate. Then it is transferred into previously sterilized ampoules, sealed properly and sterilized by autoclaving.

USE: It is used as mineral supplement and medication.

REPORT: Calcium Gluconate injection is prepared

EXPERIMENT NO: 10

PREPARATION OF ASCORBIC ACID INJECTION

AIM: To prepare and submit 2 ml ascorbic acid injection.

REQUIREMENTS: Beaker, Glass rod, Funnel, Filter paper, Ampoule

PRINCIPLE: Injections are sterile solutions, emulsions or suspensions. They are prepared by dissolving, emulsifying or suspending an active ingredient and any other substances in water for injection. Injecting is the act of giving medication by use of syringe and needle to obtain the desired therapeutic effect taking into account the patient's safety and comfort. It is suitable for those drugs that are altered or not absorbed by other methods of administration. Ascorbic Acid (vitamin C) is a water-soluble vitamin. It occurs as a white or slightly yellow crystal or powder with a light acidic taste. It is an antiscorbutic product. Ascorbic Acid injection is a clear, colourless to slightly yellow sterile solution of Ascorbic Acid in Water for Injection, for intravenous, intramuscular or subcutaneous use.

FORMULA:

Ingredients	1 Ampoule	2 Ampoules
Ascorbic Acid	0.5 gm	1 gm
Water for injection upto	2 ml	4 ml

PROCEDURE: Ascorbic acid is dissolved in water for injection in a beaker and makes upto required volume. Filter it and take 2 ml of the filtrate. Then it is transferred into previously sterilized ampoules, sealed properly and sterilized by autoclaving.

USE: It is used as anti-scurvy.

REPORT: Ascorbic Acid injection is prepared

EXPERIMENT NO: 11

PREPARATION OF PARACETAMOL SYRUP

AIM: To prepare and submit 100 ml of paracetamol syrup.

APPARATUS REQUIREMENTS: Beaker, Glass rod, Thermometer, Funnel.

PRINCIPLE: Paracetamol is one of the most popular and most commonly used analgesic and antipyretic drugs around the world, available without a prescription. It is the drug of choice in patients that cannot be treated with non-steroidal anti-inflammatory drugs (NSAID), such as people with bronchial asthma, peptic ulcer disease, salicylate-sensitized people, pregnant or breastfeeding women. It is recommended as a first-line treatment of pain associated with osteoarthritis. Paracetamol syrup is commonly used in children. But many formulations used the higher amount of sweetener that causes mostly children diabetes and use of higher amount of preservatives causes major side effects. So the use of sweetener and preservative should be as per the guideline for manufacturing related to the country.

Ingredients table (Formulation of paracetamol syrup- 250 mg /10 ml)

Ingredients	Weight	Function
PART I		
Paracetamol	1.25g	Active ingredient
Polyethylene glycol 6000 (PEG 6000)	5.0 g	Solubilizer
Glycerin	1.25 g	Diluent and sweetener
D.M. Water	15.0 ml	Diluent
PART II		
Sucrose	15.0 g	Sweetening agent
D.M Water	10.0 ml	Diluent
Propylene glycol	0.002 g	Preservative
Citric acid monohydrate	0.030 g	pH modifier

PROCEDURE:

Part I

1. Heat (PEG 6000) at 50°C and add Paracetamol in it. Stir the solution for 30 minutes.
2. Heat Glycerin at 50°C and then add in step 1 under continuous stirring. Stir the solution for 20 minutes. Transparent solution will be obtained.
3. Heat water at 50°C and put it under continuous stirring.

4. Add above solution ((PEG 6000) + Paracetamol+ Glycerin) slowly into D.M. Water under continuous stirring. The transparent solution will be obtained.

Part II

5. Weigh accurately sucrose. Add sucrose in hot (65°C) D.M. Water under continuous stirring till it dissolved.
6. Filter the above solution and keep filtrate under stirring.
7. Added Preservative, Sweetener in it with continuous stirring for 10 minutes.

Mixing of Part I and Part II

8. Slowly add part I in Part II under continuous stirring. Stir it till clear solution is obtained.
9. Check pH above solution. If pH is not between 3.80-6, then add accordingly Citric acid solution to adjust pH.
10. Add color solution in above solution under stirring.
Now, add flavor under continuous stirring
11. Make volume 100 ml of D.M. water if required.
12. Clear transparent Paracetamol syrup is obtained

REPORT: Paracetamol Syrup is prepared.

EXPERIMENT NO: 12

PREPARATION OF PHYSOSTIGMINE EYE DROPS

AIM: To prepare and submit 10 ml of Physostigmine eye drop.

REQUIREMENTS: Beaker, Glass rod, Measuring cylinder

PRINCIPLE: Eye drops are saline-containing drops used as an ocular route to administer. Depending on the condition being treated, they may contain steroids, antihistamines, sympathomimetics, nonsteroidal anti-inflammatory drugs (NSAIDs), antibiotics, antifungal, or topical anesthetics. Eye drops sometimes do not have medications in them and are only lubricating and tear-replacing solutions. Eye drops are also used for stopping itching and redness of the eyes. Physostigmine ophthalmic reduces pressure in the eye by increasing the amount of fluid that drains from the eye. It is used to treat glaucoma by lowering pressure inside the eye. Here benzalkonium chloride is used as bactericide and sodium metabisulphite is used as reducing agent.

FORMULA:

Ingredients	For 100 ml	For 10 ml
Physostigmine sulphate	0.5 gm	
sodium metabisulphite	0.2gm	
Benzalkonium chloride solution	0.02 gm	
Purified water upto	100.0 ml	

PROCEDURE: Mix sodium metabisulphite and Benzalkonium chloride solution dissolve the medicament in the mixture and adjust the final volume with purified water. Filter the solution and packed in a previously sterilized suitable container or sterilize it after packing.

PRECAUTION: Avoid contamination during use.

REPORT: Physostigmine Eye Drops are prepared.

EXPERIMENT NO: 13

PREPARATION OF ATROPINE EYE DROPS

AIM: To prepare and submit 10 ml of Atropine eye drop.

REQUIREMENTS: Beaker, Glass rod, Measuring cylinder, Conical flask

PRINCIPLE: Eye drops are saline-containing drops used as an ocular route to administer. Depending on the condition being treated, they may contain steroids, antihistamines, sympathomimetics, nonsteroidal anti-inflammatory drugs (NSAIDs), antibiotics, antifungal, or topical anesthetics. Eye drops sometimes do not have medications in them and are only lubricating and tear-replacing solutions. Eye drops are also used for stopping itching and redness of the eyes. Atropine eye drop is used before eye examinations (e.g., refraction) and to treat certain eye conditions (e.g., uveitis). It belongs to a class of drugs known as anticholinergic. Atropine works by widening (dilating) the pupil of the eye.

FORMULA:

Ingredients	For 100 ml	For 10 ml
Atropine sulphate	1 gm	
Phenyl mercuric nitrate solution, 0.004% w/v	50 ml	
Purified water upto	100 ml	

PROCEDURE: Weigh the medicament and dissolve it in the bactericidal solution in a small conical flask. Transfer it to a 10 ml measure, rinse the flask, and adjust the final volume with purified water. Sterilize it by autoclaving at 115°C for 30 mins.

PRECAUTION: Avoid contamination during use.

REPORT: Atropine Eye Drops is prepared.

EXPERIMENT NO: 14

PREPARATION OF COLD CREAM

AIM: To prepare and submit 10gms of cold cream (w/o type of emulsion)

APPARATUS: Beaker, glass rod, china dish, mortar and pestle, thermometer.

PRINCIPLE: Cold cream is w/o type of emulsion, which when applied to the skin, a cooling effect is produced, due to the slow evaporation of water, present in emulsion. Cold cream is prepared by saponification reaction between and alkali-borax; i.e borax reacts with free fatty acids of bees wax and produce borax soap in-situ (ester of fatty acid). This soap acts as emulsifying agent.

In cold cream, the internal phase is oil and external phase is water, hence it forms o/w type of emulsion. But after application on the skin, water evaporates and leads to phase inversion from o/w type to w/o type emulsion. Therefore oily phase, which is remaining (left) on the skin, gives emollient nature. Liquid paraffin is used as emollient and rose oil is used as perfume, to give a pleasant flavour to the cream.

Ingredients table (Formula):

Ingredients	Official formula	Working formula
White Bees Wax		
Liquid paraffin(emollient)		
Borax		
Water		
Perfume		

PROCEDURE:

Since there will be little wastage ((loss) during weighing and preparing, to manipulate these practical losses, calculate the ingredients for at least one or two grams extra, than prescribed.

- 1) Grate the white beeswax in to small pieces. Weigh the required quantity of white beeswax and liquid paraffin and melt in china dish, by heating on a water bath up to 70°C.
- 2) In a glass beaker, dissolve borax in water and heat up to 70°C
- 3) When both oily and aqueous phases reach the same temperature (70°C), gradually add

borax solution to the melt of beeswax, with constant stirring.

- 4) Stir continuously until it becomes cool. When the temperature lowers to 40-45°C, incorporate rose oil and mix uniformly, until a homogenous semi solid mass is obtained.

Dispensing: weigh the prescribed quantity of cream on a butter paper and transfer to an ointment jar or metallic/plastic collapsible tube, close it thoroughly and label.

DIRECTION: Apply to skin.

USES: Cold cream is used as an emollient for the treatment of dry skin. Hence this becomes quite popular in winter season.

STORAGE: Store in a cool place but do not allow to freeze.

Auxiliary label: FOR EXTERNAL USE ONLY

REPORT: Cold cream is prepared.

EXPERIMENT NO: 15

PREPARATION OF VANISHING CREAM

AIM: - To prepare and submit 10gms of vanishing cream (o/w type).

APPARATUS: China dish, glass rod, beaker, Bunsen burner, thermometer

PRINCIPLE: Vanishing cream is o/w type of emulsion, which when applied to the skin, it vanishes and leaves an almost invisible layer on it. Hence it is called as 'vanishing cream'. The layer left behind after application, acts as a base or foundation, for facial make up. Hence vanishing creams are also called as 'foundation creams'. Since water is an external phase, it will be quickly washed off with water.

The main ingredients of vanishing creams are stearic acid, alkali and water. Stearic acid gives a pearly white shining appearance to the cream, which on application gives a thin white film of free stearic acid. Soap is prepared in-situ by the chemical reaction between alkali and stearic acid, which is used as emulsifying agent.

Vanishing creams are o/w type emulsion; there is a possibility of evaporation of water from the external phase of emulsion. Therefore, glycerine, polyethylene glycol or alcohol are incorporated as humectants, to prevent the drying out of cream, since external phase of vanishing cream is aqueous, it should be protected from the contamination, from microorganisms by adding suitable preservatives, like methyl paraben or propyl paraben. These creams are also be scented pleasantly, using suitable perfumes in small quantities.

Ingredients	Official Formula(64 gm)	Working Formula
Stearic acid		
Potassium hydroxide		
Glycerine(humectants)		
Methyl paraben		
Water		
Perfume		

PROCEDURE:

- Melt stearic acid in china dish on water bath by heating up to 70⁰C. In a beaker, Dissolve KOH, and methyl paraben (methyl parahydroxybenzoate) in water, add glycerin to it.
- Heat this aqueous solution up to 70⁰C on water bath.
- When both aqueous and oil phases reaches the same temperature 70⁰C, add aqueous phase to the melted stearic acid with continuous stirring.
- Remove the dish from heat and continue the stirring and when temperature reaches 40⁰C, add perfume.
- Mix uniformly until it becomes cool and homogenous cream is obtained.

DISPENSING: Weigh the prescribed quantity of cream on the butter paper and transfer to a wide mouthed, small, screw capped plastic or glass bottle or to collapsible tube, seal and label.

DIRECTION: Used for external application. Apply to skin where ever necessary.

STORAGE: store in a cool place.

AUXILIARY LABEL: FOR EXTERNAL USE ONLY

USES: vanishing cream is used as foundation for holding the makeup preparation for longer period.

REPORT: Vanishing Cream is prepared.

EXPERIMENT NO: 16

EVALUATION OF GLASS CONTAINERS (AS PER IP)

AIM: To carryout different evaluate tests of glass container as per I.P.

REQUIREMENTS: Class container, Beaker, Conical flask, Burette, Mortar and pestle, Sieve

PRINCIPLE: Glass containers may be colourless or coloured. Neutral glass is a borosilicate glass containing significant amounts of boric oxide, aluminum oxide, alkali and/or alkaline earth oxides. It has a high hydrolytic resistance and a high thermal shock resistance. Soda-lime-silica glass is a silica glass containing alkali metal oxides, mainly sodium oxide and alkaline earth oxides, mainly calcium oxide. It has only a moderate hydrolytic resistance.

According to their hydrolytic resistance, glass containers are classified as:

- Type I glass containers which are of neutral glass, with a high hydrolytic resistance, suitable for most preparations whether or not for parenteral use.
- Type II glass containers which are usually of soda-lime- silica glass with high hydrolytic resistance resulting from suitable treatment of the surface. They are suitable for most acidic and neutral, aqueous preparations whether or not for parenteral use.
- Type III glass containers which are usually of soda- lime-silica glass with only moderate hydrolytic resistance. They are generally suitable for non-aqueous preparations for parenteral use, for powders for parenteral use and for preparations not for parenteral use.

Glass containers intended for parenteral preparations may be ampoules, vials or bottles. Glass is a common material to be used in either non sterile or sterile liquid dosage forms. It leaches alkali from its surface. Hence, a limit test for alkalinity is to be performed before using it for a particular product. USP and IP provide two tests to determine the chemical resistance of glass containers.

1. Powdered Glass Test

From the glass containers, alkaline constituents (oxides of sodium, potassium, calcium, aluminum, etc.) are leached into purified water under conditions of elevated temperatures. When the glass is powdered the leaching of alkali can be enhanced in the powdered is critical. The principle involved in the powdered glass test in estimate the amount of alkali leached form the glass powder. The amount of acid that is necessary to neutralize the released alkali (a specified limit) is specified in the pharmacopoeia. The basic analysis is acid-base titration using methyl red indicator.

2. Water Attack Test

This is only for treated soda lime glass containers under the controlled humidity conditions which neutralize the surface alkali and glass will become chemically more resistant. The principle involved in the water attack test is to determine whether the alkali leached from the surface of a container is within the specified limits or not. Since the inner surface is under test entire container (ampoule) has to be used. The amount of acid that is necessary to neutralize the released alkali from the surface is estimated, the leaching of alkali is accelerated using elevated temperature for a specified time. Methyl red indicator is used to determine the end point. The basic is acid-base titration.

PROCEDURE:

Powdered glass test:

Step-1: Preparation of glass specimen: Few containers are rinsed thoroughly with purified water and dried with stream of clean air. Grind the containers in a mortar to a fine powder and pass through sieve no.20 and 50.

Step-2: Washing the specimen: 10gm of the above specimen is taken into 250 ml conical flask and wash it with 30 ml acetone. Repeat the washing, decant the acetone and dried the specimen after which it is used within 48hr.

Step-3: 10gm sample is added with 50ml of high purity water in a 250ml flask. Place it in an autoclave at $121^{\circ}\text{C}\pm 2^{\circ}\text{C}$ for 30min. Cool it under running water. Decant the solution into another flask, wash again with 15ml high purity water and again decant. Titrate immediately with 0.02N sulphuric acid using methyl red as an indicator and record the volume.

Water attack test:

Rinse thoroughly with high purity water. Fill each container to 90% of its overflow capacity with water and is autoclaved at 121°C for 30min then it is cooled and the liquid is decanted which is titrated with 0.02N sulphuric acid using methyl red as an indicator. The volume of sulfuric acid consumed is the measure of the amount of alkaline oxides present in the glass containers.

Limits of alkalinity for glass containers:

TESTS	CONTAINER	VOL. OF 0.02N H₂SO₄
Powdered glass test	Type I	
	Type II	
	Type III	
Water attack test	Type II(100ml or below)	
	Type II(above 100ml)	

REPORT: Glass container is evaluated.