

Continue Learning with FirstHope

Complete Notes on the FirstHope App

Complete subject and semesters are available on FirstHope app only.

- Get complete subject and semester notes in the FirstHope app.
- Available for B.Pharm, D.Pharm, M.Pharm and Pharm.D students.

[Click here to get the FirstHope app](#)

Join the FirstHope Telegram Channel

Join FirstHope Notes and keep up with channel updates.

[Click here to join Telegram](#)

Instagram

@firsthope.official

[Click here to follow Instagram](#)

YouTube Channel

FirstHope-Pharmacy / Live streams

[Click here to watch the YouTube channel](#)

WhatsApp Channel

Follow FirstHope on WhatsApp.

[Click here to join the WhatsApp channel](#)

FirstHope Website

firsthopenotes.com

[Click here to visit the website](#)

Semester 2 - Practical Notes

Physical Pharmaceutics

Complete Subject Manual

15 practical files and activities. Use the PDF bookmarks to navigate. Page numbers restart within each practical; content page counts below exclude promotion pages.

Contents

No.	Practical	Content pages
1	Surface Tension by Drop Count and Drop Weight Methods	2
2	Critical Micellar Concentration of a Surfactant	2
3	Viscosity by Ostwald and Brookfield Viscometers	2
4	HLB Value and Surfactant-Blend Evaluation	2
5	Isotonicity by Sodium Chloride Equivalent and White-Vincent Methods	2
6	Particle Size Distribution by Sieving	2
7	Particle Size Distribution by Microscopy	2
8	Bulk Tapped and True Density with Derived Powder Properties	2
9	Angle of Repose and Effect of Glidants	2
10	Drug Solubility in Different pH and Buffer Systems	2
11	Partition Coefficient in n-Octanol and Water	2
12	Partition Coefficient in Benzene and Water	2
13	Critical Solution Temperature and Composition of Phenol-Water	2
14	Apparent Specific Surface Area of Charcoal by Acetic-Acid Adsorption	2
15	Buffer Capacity and pKa During Weak-Acid Titration	2

Surface Tension by Drop Count and Drop Weight Methods

Experiment 1 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To estimate liquid surface tension relative to a reference using both drop methods.

Principle

Drop detachment balances capillary forces and drop weight. Relative methods use the same clean tip and controlled drop formation. The simple ratios assume comparable drop correction; differences in tip wetting, flow and surface age can affect the result.

Requirements

- Clean stalagmometer/drop apparatus, test liquid, reference liquid and their densities at the test temperature.
- Temperature control, stopwatch, weighed collection vessels and balance.

Procedure

1. Condition

Clean and rinse the tip with the appropriate liquid, position it vertically and equilibrate both liquids to the same recorded temperature.

2. Count reference

Count drops between the two fixed volume marks for the reference. Repeat at least three times and report the mean.

3. Count test liquid

Repeat over the same marked volume and controlled flow conditions. Avoid bubbles and partial starting/ending drops.

4. Drop-weight reference

Collect a known number of complete reference drops in a covered weighed vessel. Obtain total net mass and mean mass per drop.

5. Drop-weight test

Repeat for the test liquid with the same tip and comparable formation conditions. Limit evaporation.

6. Calculate

Use the appropriate ratio below. Include density in the fixed-volume drop-count ratio, but do not multiply the measured drop-mass ratio by density again.

Observations and Record

Method	Reference / test records	Calculated value
Drop count	$N_r = \text{___}$; $N_x = \text{___}$; densities = ___	$\gamma_x = \text{___}$ mN/m
Drop weight	Net mass / number of drops = ___	$\gamma_x = \text{___}$ mN/m
Conditions	Temperature / tip / formation rate	_____

Worked Example

The numbers below are illustrative; enter your own observations in the record.

- Drop count: $\gamma_x = \gamma_r \times (\rho_x N_r) / (\rho_r N_x)$.
- Illustrative assigned $\gamma_r = 72.0$ mN/m, $\rho_x/\rho_r = 0.90$, $N_r = 50$ and $N_x = 60$: $\gamma_x = 54.0$ mN/m.
- Separate drop-weight illustration: $\gamma_x = \gamma_r \times \text{mean } m_x / \text{mean } m_r$. With 0.040 g and 0.050 g, $\gamma_x = 57.6$ mN/m. These are separate datasets.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use the instructor's assigned material and apparatus; record actual temperature and units with every measured property.
- Check instrument calibration and use repeat readings rather than a single convenient result.
- Compare a marketed teaching formulation only under the same method and conditions; its excipients can change the property.
- Use the reference surface tension appropriate to the actual temperature; surfactant solutions can depend on drop formation time.

Viva Questions and Answers

1. Why use the same tip?

Capillary geometry affects detachment.

2. Why does drop count use density?

Mass per fixed volume depends on liquid density.

3. Why avoid rapid drops?

Formation conditions and surface adsorption may change the measured value.

Critical Micellar Concentration of a Surfactant

Experiment 2 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To estimate the CMC from a change in a measured property across surfactant concentrations.

Principle

Below the CMC, increasing concentration mainly changes the population of individual surfactant molecules. Above it, micelles form and the concentration dependence of conductivity or surface tension changes. CMC depends on temperature, electrolyte content and the chosen surfactant.

Requirements

- Assigned ionic surfactant for conductivity, or a validated surface-tension method.
- Accurately prepared concentration series, purified water, calibrated meter/tensiometer and temperature control.

Procedure

1. Plan range

Prepare concentrations spanning the expected transition, with closely spaced points near it. Record concentration units, purity and any added electrolyte.

2. Blank and calibrate

Check the chosen instrument with its prescribed standard and water blank. Maintain one temperature throughout.

3. Measure

Measure each solution after the same equilibration procedure. Rinse the cell correctly between solutions and take repeat readings.

4. Plot

For conductivity, plot conductivity against molar concentration. For surface tension, plot surface tension against $\log_{10}$ concentration.

5. Fit branches

Fit the approximately linear pre- and post-transition regions without forcing all points into one line. Find their intersection.

6. Report

Convert the intersection to the original concentration units. Give the method, temperature, range and uncertainty/replicate spread; a weak or broad transition requires qualification.

Observations and Record

Concentration (mol/L)	Property and units	Repeat / mean
_____	_____	_____
_____	_____	_____
_____	_____	_____

Worked Example

The numbers below are illustrative; enter your own observations in the record.

- If a surface-tension intersection is at $\log_{10} C = -2.10$, $CMC = 10^{(-2.10)} = 0.00794 \text{ mol/L} = 7.94 \text{ mmol/L}$.
- This constructed example is not a universal CMC for every surfactant or water composition.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use the instructor's assigned material and apparatus; record actual temperature and units with every measured property.
- Check instrument calibration and use repeat readings rather than a single convenient result.
- Compare a marketed teaching formulation only under the same method and conditions; its excipients can change the property.

Viva Questions and Answers

1. Why use logarithmic concentration for a surface-tension plot?

It often separates the pre-transition trend clearly over a wide concentration range.

2. Does added salt leave CMC unchanged?

No. Electrolytes can substantially affect ionic surfactants.

3. Can conductivity measure every non-ionic surfactant well?

Not necessarily; choose a suitable validated property method.

Viscosity by Ostwald and Brookfield Viscometers

Experiment 3 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To compare relative capillary viscosity and rotational apparent viscosity.

Principle

An Ostwald viscometer relates capillary flow time to viscosity for suitable Newtonian liquids. A Brookfield instrument measures resistance to spindle rotation; a non-Newtonian material can give different apparent viscosities at different speeds.

Requirements

- Ostwald viscometer, Brookfield instrument and appropriate reference standard.
- Test liquid, density data, bath, timing device and the prescribed spindle/container.

Procedure

1. Temperature

Equilibrate liquids and instruments at one recorded temperature. Check that the chosen material is suitable for each method.

2. Ostwald reference

Fill to the specified level without bubbles. Draw liquid above the upper timing mark with a safe aid and time flow between marks. Repeat.

3. Ostwald test

Measure test-liquid flow times and density under the same conditions. Reject disturbed flow or air bubbles.

4. Brookfield setup

Level and check the instrument. Fit the specified spindle and immerse to its mark in the prescribed container, avoiding trapped air.

5. Rotational readings

Choose a spindle/speed combination within the manufacturer's usable torque range. Record spindle, rpm, torque, temperature and elapsed time for each reading.

6. Interpret

Calculate capillary viscosity from the ratio and report rotational values with their conditions. Do not call a speed-dependent reading a universal constant.

Physical Pharmaceutics

Observations and Record

Method	Raw record	Report
Ostwald	Mean t_r / t_x ; ρ_r / ρ_x	$\eta_x = \text{___ mPa}\cdot\text{s}$
Brookfield	Spindle / rpm / torque / time	Apparent $\eta = \text{___ mPa}\cdot\text{s}$
Comparison	Same temperature and preparation?	_____

Worked Example

The numbers below are illustrative; enter your own observations in the record.

- $\eta_x = \eta_r \times \rho_x t_x / (\rho_r t_r)$.
- For an assigned reference $\eta_r = 1.00 \text{ mPa}\cdot\text{s}$, $\rho_r = 1.00 \text{ g/mL}$, $t_r = 60 \text{ s}$, $\rho_x = 1.10 \text{ g/mL}$ and $t_x = 180 \text{ s}$, $\eta_x = 3.30 \text{ mPa}\cdot\text{s}$.
- The reference values are illustrative; use its certified value at the actual bath temperature. $1 \text{ cP} = 1 \text{ mPa}\cdot\text{s}$.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use the instructor's assigned material and apparatus; record actual temperature and units with every measured property.
- Check instrument calibration and use repeat readings rather than a single convenient result.
- Compare a marketed teaching formulation only under the same method and conditions; its excipients can change the property.

Viva Questions and Answers

1. Why control temperature closely?

Viscosity is temperature dependent.

2. What is apparent viscosity?

The value obtained under specified flow/shear conditions for a material whose behaviour may depend on them.

3. Why record torque?

An unsuitable torque range can make a reading unreliable.

HLB Value and Surfactant-Blend Evaluation

Experiment 4 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To calculate a suitable HLB value and evaluate a series of surfactant blends.

Principle

Hydrophilic-lipophilic balance describes relative affinity for water and oil within a stated system. Griffin's mass expression applies to suitable non-ionic surfactants; it is not a universal formula for every surfactant. A blend's HLB is approximated by a mass-weighted average.

Requirements

- Assigned surfactants with supplier HLB data, selected oil and identical teaching emulsion compositions.
- Balance, mixing setup, containers and the laboratory stability-observation method.

Procedure

1. Identify approach

For an appropriate non-ionic molecule with known hydrophilic mass M_h and total molar mass M , calculate $HLB = 20 M_h/M$. Otherwise use the applicable method or supplier value.

2. Plan blends

For surfactants A and B, $HLB_{blend} = f_A HLBA + (1-f_A) HLBB$. Keep total surfactant mass and oil/water composition constant.

3. Prepare series

Weigh several blends covering the assigned HLB range and prepare emulsions by one controlled mixing method.

4. Observe

Record emulsion type, appearance, creaming and separation at stated times. A conductivity/dilution test can help assess emulsion type.

5. Compare

Identify the most suitable blend for the chosen oil and test conditions. This estimates that system's required HLB rather than a new intrinsic HLB for an unknown ingredient.

6. Report

List actual component values, fraction calculation, composition and observation interval. Do not equate a short-term appearance with validated shelf life.

Physical Pharmaceutics**Observations and Record**

Blend HLB	A / B masses	Emulsion type / stability record
_____	_____	_____
_____	_____	_____
_____	_____	_____

Worked Example

The numbers below are illustrative; enter your own observations in the record.

- If $M_h = 300$ and $M = 600$ g/mol for a suitable non-ionic surfactant, Griffin HLB = 10.0.
- Using assigned Tween 80 HLB 15.0 and Span 80 HLB 4.3, target HLB 10.0 requires fTween = $(10.0-4.3)/(15.0-4.3) = 0.5327$.
- For a 1.000 g blend, use 0.5327 g Tween 80 and 0.4673 g Span 80, subject to the actual supplier data.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use the instructor's assigned material and apparatus; record actual temperature and units with every measured property.
- Check instrument calibration and use repeat readings rather than a single convenient result.
- Compare a marketed teaching formulation only under the same method and conditions; its excipients can change the property.

Viva Questions and Answers**1. What does required HLB refer to?**

The HLB range that best emulsifies a particular oil system under defined conditions.

2. Does the blend equation use mass or volume fraction?

Mass fraction.

3. Can the Griffin expression be applied blindly to ionic surfactants?

No. Its applicability must be checked.

Isotonicity by Sodium Chloride Equivalent and White-Vincent Methods

Experiment 5 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To calculate an isotonicity adjustment for an educational formulation.

Principle

A sodium chloride equivalent E is the mass of NaCl osmotically equivalent to 1 g of a solute under the stated assumptions. The calculations estimate tonicity contributions; they do not establish sterility, compatibility or clinical suitability.

Requirements

- Teacher-provided hypothetical formulation, final volume and verified E values.
- Calculator, calculation sheet and, if demonstrated, labelled non-clinical teaching materials.

Procedure

1. List components

Record each solute mass in grams and its applicable E value. Include every relevant solute and check concentration-dependent assumptions.

2. NaCl target

For a 0.9% w/v NaCl target, calculate $0.009 \times \text{final volume in mL}$, expressed in grams.

3. Subtract contribution

Calculate $\sum wE$ and subtract it from the NaCl target. A negative result indicates the formulation is already above that target; adding salt cannot correct it.

4. White-Vincent volume

Calculate the approximate volume made isotonic by the solutes: $V_{iso} = 111.1 \sum wE \text{ mL}$.

5. Completion

For the White-Vincent approach, conceptually add water to V_{iso} and then the appropriate isotonic vehicle to final volume. The method and vehicle must be approved for the actual formulation.

6. Check

Confirm units, final volume and arithmetic. State that these are teaching calculations rather than preparation instructions for patient use.

Physical Pharmaceutics**Observations and Record**

Component	Mass w (g)	E and wE (g NaCl equivalent)
Assigned drug	_____	_____
Other solutes	_____	_____
Final volume / target / adjustment	_____	_____

Worked Example

The numbers below are illustrative; enter your own observations in the record.

- For 0.200 g drug with $E = 0.18$ in 20.0 mL: $wE = 0.0360$ g.
- $\text{NaCl needed} = 0.009 \times 20.0 - 0.0360 = 0.1440$ g.
- White-Vincent volume = $111.1 \times 0.0360 = 4.00$ mL, followed by approved isotonic vehicle to 20.0 mL.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use E values applicable to the actual chemical form and method.
- Keep grams and millilitres consistent and do not treat a negative salt requirement as permission to use a negative mass.
- Educational calculations alone do not authorise sterile product preparation or dispensing.

Viva Questions and Answers**1. What does $E = 0.18$ mean?**

One gram of that solute has an approximate NaCl-equivalent contribution of 0.18 g under the stated method.

2. Why sum all solutes?

Each relevant solute contributes to osmotic effects.

3. What if the NaCl requirement is negative?

The composition requires reconsideration; adding NaCl would increase the excess.

Particle Size Distribution by Sieving

Experiment 6 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To determine a mass-based size distribution of a powder using a sieve stack.

Principle

Particles are separated by their ability to pass defined openings. The result is a mass distribution of sieve fractions, influenced by particle shape, agglomeration and sieving conditions. It is different from a number-based microscopy distribution.

Requirements

- Dry representative teaching powder, verified sieve openings and collection pan.
- Balance, sieve shaker/timer, brush and appropriate dust control.

Procedure

1. Prepare

Record the initial powder mass, moisture condition and sieve openings in micrometres. Assemble largest opening at the top and a pan below.

2. Sieve

Load a suitable quantity, close the stack and shake for the prescribed time/amplitude. Avoid overloading and forcing particles through openings.

3. Weigh fractions

Weigh retained material on each sieve and in the pan. Account for material loss and record recovered total.

4. Calculate

Calculate each fraction percentage using the stated denominator. Compare recovered and initial masses to check the mass balance.

5. Graph

Plot percent retained and/or cumulative percent undersize against a clearly defined size axis. State any logarithmic scale.

6. Mean size

For bounded fractions, use assigned representative sizes such as arithmetic midpoints. Open-ended top/pan fractions need justified bounds before a mean is calculated.

Observations and Record

Bounded interval (µm)	Mass / % retained	Representative d (µm)
300-500	20 g / 20%	400
150-300	50 g / 50%	225
75-150	30 g / 30%	112.5

Worked Example

The numbers below are illustrative; enter your own observations in the record.

- Illustrative 100 g dataset with all material inside the listed bounded intervals: $d_{mass} = \sum m_i d_i / \sum m_i$.
- $d_{mass} = (20 \times 400 + 50 \times 225 + 30 \times 112.5) / 100 = 226.25 \mu\text{m}$.
- If material also occurs above 500 µm or below 75 µm, report those fractions and revise the calculation; do not assign an unjustified pan diameter.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use the instructor's assigned material and apparatus; record actual temperature and units with every measured property.
- Check instrument calibration and use repeat readings rather than a single convenient result.
- Compare a marketed teaching formulation only under the same method and conditions; its excipients can change the property.
- Minimise airborne powder; do not brush strongly against or damage sieve meshes.

Viva Questions and Answers

1. Why check recovered mass?

Loss can distort the percentage distribution.

2. What kind of average is this?

A mass-weighted average using assumed representative fraction sizes.

3. Why can elongated particles mislead?

Passage depends on orientation as well as nominal dimensions.

Particle Size Distribution by Microscopy

Experiment 7 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To obtain a calibrated number-based particle size distribution.

Principle

Microscopy measures a stated projected particle dimension. An eyepiece scale must be calibrated at the objective used. Counting particles gives a number distribution; it must not be mistaken for a mass or volume distribution.

Requirements

- Representative powder dispersion, microscope, stage micrometer and eyepiece scale or calibrated imaging system.
- Slides, coverslips, suitable non-dissolving dispersant and counting worksheet.

Procedure

1. Calibrate

Align stage and eyepiece scales at the chosen objective. Calculate micrometres per division and repeat calibration when the objective or image scaling changes.

2. Disperse

Prepare a thin random dispersion without dissolving particles or breaking them unintentionally. Avoid overlapping agglomerates.

3. Define dimension

Choose a projected dimension, for example a stated Feret diameter, and use it consistently for all particles.

4. Count systematically

Use a fixed field-selection and boundary rule to avoid preferentially selecting large particles. Measure the assigned number, commonly at least 100 in teaching work.

5. Group

Place measured dimensions into defined non-overlapping classes and calculate count percentages and cumulative counts.

6. Report

Calculate number mean where appropriate and state the dispersion method, count, calibration and dimension definition.

Physical Pharmaceutics**Observations and Record**

Size class / representative d	Particle count n	n × d
20 µm teaching group	5	100 µm
40 µm teaching group	5	200 µm
Total	10	300 µm

Worked Example

The numbers below are illustrative; enter your own observations in the record.

- If 100 µm on a stage micrometer matches 25 eyepiece divisions, calibration = 4.00 µm/division.
- For the short illustrative dataset above, number mean = $300/10 = 30.0$ µm. Use the full required count for the actual practical.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use the instructor's assigned material and apparatus; record actual temperature and units with every measured property.
- Check instrument calibration and use repeat readings rather than a single convenient result.
- Compare a marketed teaching formulation only under the same method and conditions; its excipients can change the property.

Viva Questions and Answers**1. Why recalibrate for each objective?**

Image magnification changes the size represented by one scale division.

2. Is the number mean the same as the sieve mass mean?

No. Large particles contribute far more mass but only one count each.

3. Why state a boundary rule?

It avoids counting the same particle twice in adjoining fields.

Bulk Tapped and True Density with Derived Powder Properties

Experiment 8 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To determine powder densities, Hausner ratio, Carr index and bed porosity.

Principle

Bulk and tapped densities include voids between particles; true density measures solid material volume by the assigned displacement method. Tapping rearranges particles. Derived indices depend on the complete method and do not uniquely establish powder-flow behaviour.

Requirements

- Representative dry powder, calibrated cylinder and approved tapping apparatus.
- Balance and validated pycnometer/displacement method with a non-reactive, non-dissolving displacement medium.

Procedure

1. Bulk volume

Weigh the assigned powder mass and introduce it gently without intentional compaction. Read the initial bed volume V_0 .

2. Tapped volume

Tap using the prescribed apparatus and current laboratory/compendial sequence until its endpoint criterion is satisfied. Record tap counts and final volume V_f .

3. True volume

Use the assigned calibrated displacement procedure, controlling temperature and removing trapped air without dissolving or reacting with the powder.

4. Densities

Calculate bulk m/V_0 , tapped m/V_f and true m/V_{solid} , retaining consistent units.

5. Indices

Calculate Hausner ratio, Carr compressibility index and bulk-bed porosity. State whether the true-density method includes or excludes internal accessible pores.

6. Compare

Report replicate spread and method conditions. Interpret flow-related indices alongside actual flow testing rather than assigning an unsupported universal grade.

Physical Pharmaceutics**Observations and Record**

Record	Value
Mass / V_0 / V_f / tap endpoint	_____
Solid displacement volume / method	_____
Bulk / tapped / true density	_____
Hausner / Carr index / porosity	_____

Worked Example

The numbers below are illustrative; enter your own observations in the record.

- For $m = 50.0$ g, $V_0 = 100$ mL and $V_f = 80$ mL: $\rho_{\text{bulk}} = 0.500$ g/mL; $\rho_{\text{tap}} = 0.625$ g/mL.
- Hausner = $\rho_{\text{tap}}/\rho_{\text{bulk}} = V_0/V_f = 1.25$. Carr index = $100(V_0 - V_f)/V_0 = 20.0\%$.
- With assigned $\rho_{\text{true}} = 1.50$ g/mL, bulk-bed porosity = $(1 - \rho_{\text{bulk}}/\rho_{\text{true}}) \times 100 = 66.7\%$.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use the instructor's assigned material and apparatus; record actual temperature and units with every measured property.
- Check instrument calibration and use repeat readings rather than a single convenient result.
- Compare a marketed teaching formulation only under the same method and conditions; its excipients can change the property.

Viva Questions and Answers**1. Why is tapped density usually higher?**

Tapping reduces interparticle void volume.

2. Does bulk density equal true density?

No. Bulk volume includes bed voids.

3. Why state the displacement method?

Different accessible pores and solvent interactions can affect the measured volume.

Angle of Repose and Effect of Glidants

Experiment 9 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To compare powder-pile angle with and without an assigned glidant.

Principle

A powder cone's height and base radius define its repose angle under the chosen formation method. Friction, cohesion, particle size and humidity influence the result. A glidant can alter the measured flow pattern at a suitable concentration.

Requirements

- Assigned powder and glidant concentration, for example a supervised 0.5% w/w teaching blend.
- Fixed funnel setup, flat base, ruler, balance and mixing vessel.

Procedure

1. Control setup

Fix the funnel geometry, outlet-to-base position, powder amount, base surface and room conditions. Record whether height is fixed or adjusted by the prescribed method.

2. Untreated powder

Allow powder to form a cone without vibration or external pushing. Measure height h and two perpendicular base diameters.

3. Calculate radius

Use half the mean diameter as radius r . Repeat pile formation and record each result.

4. Prepare blend

Weigh the prescribed glidant and mix by a controlled method. Keep the same total test mass and setup.

5. Repeat

Form replicate cones for the treated powder and record h , r and any irregularity or failure to flow.

6. Compare

Calculate $\theta = \arctan(h/r)$ in degrees. Compare conditions and replicate spread; do not assume that more glidant always improves flow.

Physical Pharmaceutics**Observations and Record**

Powder condition	h / r (same units)	Angle θ (degrees)
Without glidant	_____	_____
With glidant	_____	_____
Replicate / room conditions	_____	_____

Worked Example

The numbers below are illustrative; enter your own observations in the record.

- Untreated $h = 3.0$ cm and $r = 5.0$ cm gives $\theta = \arctan(0.60) = 30.96^\circ$.
- Treated $h = 2.5$ cm and $r = 5.0$ cm gives $\theta = 26.57^\circ$ under the same method.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use the instructor's assigned material and apparatus; record actual temperature and units with every measured property.
- Check instrument calibration and use repeat readings rather than a single convenient result.
- Compare a marketed teaching formulation only under the same method and conditions; its excipients can change the property.

Viva Questions and Answers**1. Why use radius rather than diameter?**

The cone geometry uses h/r .

2. Why control humidity?

Moisture can change interparticle adhesion.

3. Does one lower angle prove all processing problems are solved?

No. Other flow, segregation and compaction properties still matter.

Drug Solubility in Different pH and Buffer Systems

Experiment 10 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To measure equilibrium solubility at controlled pH and temperature.

Principle

For ionisable compounds, total aqueous solubility can change with pH because ionised forms contribute to the dissolved concentration. The ideal relationship assumes an appropriate solid form and controlled conditions; salt formation, degradation and activity effects can cause departures.

Requirements

- Assigned teaching drug, validated buffers and concentration-assay method.
- Temperature-controlled mixing, calibrated pH meter, suitable separation method and standards.

Procedure

1. Prepare buffers

Use the assigned pH range and controlled buffer/ionic-strength conditions. Measure actual pH and temperature.

2. Add excess solid

Add sufficient drug to leave visible undissolved solid at equilibrium. Prepare replicates and a drug-free buffer blank.

3. Equilibrate

Mix for the validated period. Confirm equilibrium with consistent concentrations at successive time points rather than assuming any fixed short time is sufficient.

4. Separate

Remove undissolved material using the validated centrifugation/filtration method without cooling, adsorption loss or pH change.

5. Assay

Dilute the clear phase as needed and measure concentration against matrix-appropriate standards. Confirm that the result represents intact drug.

6. Report

Record final pH, temperature, residual solid and concentration with units. Compare marketed material only with its excipient and solid-form differences stated.

Observations and Record

Initial / final pH	Temperature / equilibration check	Solubility and units
_____	_____	_____
_____	_____	_____
_____	_____	_____

Worked Example

The numbers below are illustrative; enter your own observations in the record.

- Ideal monoprotic weak acid: $S = S_0[1 + 10^{(pH-pK_a)}]$. Weak base: $S = S_0[1 + 10^{(pK_a-pH)}]$.
- For a weak acid with $S_0 = 0.10$ mg/mL, $pK_a = 4.0$ and $pH = 5.0$, ideal $S = 1.10$ mg/mL.
- A measured 15.0 mg dissolved in 50.0 mL corresponds to 0.300 mg/mL; experimental results need not equal the ideal estimate.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use the instructor's assigned material and apparatus; record actual temperature and units with every measured property.
- Check instrument calibration and use repeat readings rather than a single convenient result.
- Compare a marketed teaching formulation only under the same method and conditions; its excipients can change the property.

Viva Questions and Answers

1. Why require excess solid?

It helps ensure the measured solution is saturated rather than merely the amount added.

2. Why measure final pH?

Drug dissolution can change the buffer pH.

3. Why validate filtration?

The filter may adsorb drug or allow fine solids through.

Partition Coefficient in n-Octanol and Water

Experiment 11 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To calculate the phase concentration ratio for a drug in n-octanol and water.

Principle

For a neutral species at equilibrium, $P = C_{\text{organic}}/C_{\text{water}}$. If ionisation contributes at a stated pH, the ratio of total analytical concentrations is a distribution ratio $D(\text{pH})$. Temperature, pH and phase composition must be controlled.

Requirements

- Assigned neutral teaching solute and staff-approved shake-flask setup, or supplied equilibrium data.
- Matched phase-assay standards, recorded phase volumes, temperature and aqueous pH.

Procedure

1. Condition phases

For the bench method approved by the laboratory, mutually pre-saturate the phases and record actual phase volumes. For a supplied-data exercise, read these conditions from the staff record.

2. Establish equilibrium

Use the staff-approved controlled mixing and settling method with the assigned solute loading. Avoid persistent emulsion and container adsorption.

3. Identify layers

Verify layer identity from the prescribed check; do not rely only on an assumed solvent order.

4. Measure both phases

Determine concentrations using phase-appropriate calibration and dilution factors. Do not substitute raw absorbance ratios from different solvent matrices.

5. Check mass balance

Calculate $C_{\text{organic}} V_{\text{org}} + C_{\text{water}} V_{\text{water}}$ and compare with initial solute mass. Investigate unexplained loss before accepting the ratio.

6. Report

Calculate P or D as applicable and $\log_{10}$ of that ratio. State phase volumes, temperature, pH, ionisation assumption and replicate agreement.

Physical Pharmaceutics**Observations and Record**

Record	Entry
Organic / aqueous phase volumes	_____
Concentrations after dilution correction	_____
Initial / recovered total solute mass	_____
P or D(pH) / $\log_{10}$ value	_____

Worked Example

The numbers below are illustrative; enter your own observations in the record.

- For $C_o = 4.00$ mg/mL and $C_w = 0.500$ mg/mL, $P = 8.00$ and $\log_{10} P = 0.9031$.
- At 10.0 mL in each phase, recovered mass = $4.00 \times 10.0 + 0.500 \times 10.0 = 45.0$ mg. Compare with an assigned initial 45.0 mg.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use the instructor's assigned material and apparatus; record actual temperature and units with every measured property.
- Check instrument calibration and use repeat readings rather than a single convenient result.
- Compare a marketed teaching formulation only under the same method and conditions; its excipients can change the property.

Viva Questions and Answers**1. Why measure both phases?**

It supports both the ratio and a mass-balance check.

2. When should the result be called D?

When the ratio concerns total species at a stated pH and ionisation affects distribution.

3. Is the ratio obtained in one solvent transferable to another?

No. The solvent system defines the partition behaviour.

Partition Coefficient in Benzene and Water

Experiment 12 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To calculate the phase concentration ratio for a drug in benzene and water.

Principle

For a neutral species at equilibrium, $P = C_{\text{organic}}/C_{\text{water}}$. If ionisation contributes at a stated pH, the ratio of total analytical concentrations is a distribution ratio $D(\text{pH})$. Temperature, pH and phase composition must be controlled.

Requirements

- Instructor-provided equilibrium records or a sealed staff demonstration. Benzene is carcinogenic; students use supplied data rather than independently preparing an open experiment.
- Matched phase-assay standards, recorded phase volumes, temperature and aqueous pH.

Procedure

1. Condition phases

For the bench method approved by the laboratory, mutually pre-saturate the phases and record actual phase volumes. For a supplied-data exercise, read these conditions from the staff record.

2. Establish equilibrium

Use the staff-approved controlled mixing and settling method with the assigned solute loading. Avoid persistent emulsion and container adsorption.

3. Identify layers

Verify layer identity from the prescribed check; do not rely only on an assumed solvent order.

4. Measure both phases

Determine concentrations using phase-appropriate calibration and dilution factors. Do not substitute raw absorbance ratios from different solvent matrices.

5. Check mass balance

Calculate $C_{\text{organic}} V_{\text{org}} + C_{\text{water}} V_{\text{water}}$ and compare with initial solute mass. Investigate unexplained loss before accepting the ratio.

6. Report

Calculate P or D as applicable and $\log_{10}$ of that ratio. State phase volumes, temperature, pH, ionisation assumption and replicate agreement.

Physical Pharmaceutics**Observations and Record**

Record	Entry
Organic / aqueous phase volumes	_____
Concentrations after dilution correction	_____
Initial / recovered total solute mass	_____
P or D(pH) / $\log_{10}$ value	_____

Worked Example

The numbers below are illustrative; enter your own observations in the record.

- For a neutral teaching solute with $C_{\text{benzene}} = 0.400 \text{ mg/mL}$ and $C_{\text{water}} = 0.200 \text{ mg/mL}$, $P = 2.00$ and $\log_{10} P = 0.3010$.
- With 10.0 mL in each phase, recovered mass = 6.00 mg. A value from this system is not an n-octanol/water $\log P$.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use the instructor's assigned material and apparatus; record actual temperature and units with every measured property.
- Check instrument calibration and use repeat readings rather than a single convenient result.
- Compare a marketed teaching formulation only under the same method and conditions; its excipients can change the property.

Viva Questions and Answers**1. Why measure both phases?**

It supports both the ratio and a mass-balance check.

2. When should the result be called D?

When the ratio concerns total species at a stated pH and ionisation affects distribution.

3. Is the ratio obtained in one solvent transferable to another?

No. The solvent system defines the partition behaviour.

Critical Solution Temperature and Composition of Phenol-Water

Experiment 13 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To construct a miscibility curve and estimate the upper critical solution temperature and composition.

Principle

A partially miscible binary system can form two liquid phases below its coexistence boundary. The upper critical solution temperature is the maximum of the miscibility curve, where the two coexisting phases become indistinguishable. Impurities and composition errors can shift the curve.

Requirements

- Instructor-prepared phenol-water composition series in the designated supervised setup, or supplied transition records.
- Controlled water bath, temperature probe, gentle mixing method and plotting worksheet.

Procedure

1. Record composition

Use mass percent phenol: $100 \frac{m_{\text{phenol}}}{m_{\text{phenol}} + m_{\text{water}}}$. Do not replace mass percent with volume percent.

2. Heat slowly

Staff supervise controlled bath heating and mixing. Record the temperature at which a cloudy two-phase mixture becomes one clear phase.

3. Cool slowly

Record the temperature of renewed turbidity while cooling under the method's mixing conditions. Repeat as prescribed.

4. Transition value

Use the defined heating/cooling evaluation method and note hysteresis. A mean is only used where that is the prescribed treatment.

5. Plot

Plot transition temperature against phenol mass percent and draw a smooth supported coexistence boundary.

6. Estimate apex

Read the highest temperature and its composition. Mark the one-phase and two-phase regions and do not force a sparse dataset to an exact literature number.

Physical Pharmaceutics**Observations and Record**

Phenol mass %	Heating / cooling transition (°C)	Evaluated transition
_____	_____	_____
_____	_____	_____
_____	_____	_____

Worked Example

The numbers below are illustrative; enter your own observations in the record.

- A constructed plot with a maximum at about 66.8°C and roughly one-third phenol by mass illustrates an upper CST. Actual composition and temperature are read from the measured curve.
- For 3.40 g phenol plus 6.60 g water, composition = 34.0% w/w phenol.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Phenol is corrosive and systemically hazardous. Use only the supervised staff-approved setup and avoid skin contact.
- No open flame, improvised sealed heating or unsupervised mixture preparation.
- Allow thermal equilibration and record both heating and cooling behaviour.

Viva Questions and Answers**1. What is an upper CST?**

The maximum coexistence temperature above which the two liquids are miscible at all compositions within the system.

2. Why measure composition by mass?

The required phase diagram uses mass fraction, which differs from volume fraction.

3. Why can impurities shift the curve?

They change component interactions and phase equilibria.

Apparent Specific Surface Area of Charcoal by Acetic-Acid Adsorption

Experiment 14 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To measure acid adsorption and estimate a model-dependent apparent surface area.

Principle

The drop in equilibrium acid concentration gives adsorbed amount per gram. A suitable Langmuir analysis can estimate monolayer capacity. Conversion to surface area assumes a molecular cross-sectional area and monolayer adsorption; this liquid-phase estimate is not a direct BET measurement.

Requirements

- Assigned activated charcoal batch, acetic-acid concentration series and standardised sodium hydroxide.
- Closed mixing vessels, filters, volumetric glassware, balance and prescribed titration endpoint method.

Procedure

1. Prepare controls

Prepare known initial acid concentrations C_0 and liquid volume V . Run no-charcoal and charcoal/leachate blanks as prescribed.

2. Adsorb

Add equal accurately weighed dry charcoal mass m to each solution, mix for the validated equilibration period and control temperature.

3. Separate

Remove charcoal by a validated method and retain clear equilibrium liquid without further adsorption loss.

4. Titrate

Determine C_e from a known aliquot using standardised base and blank correction. Acetic acid and NaOH react in a 1:1 molar ratio.

5. Calculate q

Use $q = (C_0 - C_e)V/m$ with C in mol/L, V in L and m in g. Plot the isotherm and check whether the selected model is appropriate.

6. Estimate area

For an acceptable Langmuir representation, plot C_e/q against C_e : slope = $1/qm$. Use $S = qm NA a$ with the explicitly assumed molecular area a . Report the model and limitations.

Physical Pharmaceutics

Observations and Record

C_0 / C_e (mol/L)	Charcoal m (g) / V (L)	q (mol/g) / C_e/q
_____	_____	_____
_____	_____	_____
_____	_____	_____

Worked Example

The numbers below are illustrative; enter your own observations in the record.

- A 10.0 mL equilibrium aliquot consuming 2.00 mL of 0.0500 mol/L NaOH gives $C_e = 0.0100$ mol/L after applicable blank correction.
- For $C_0 = 0.0200$, $C_e = 0.0100$, $V = 0.0500$ L and $m = 0.500$ g, $q = 0.00100$ mol/g.
- An illustrative fitted slope of 500 g/mol gives $qm = 0.00200$ mol/g.
- With an explicitly assumed $a = 0.21 \text{ nm}^2 = 0.21 \times 10^{-18} \text{ m}^2$ per molecule, $S = 0.00200 \times 6.02214076 \times 10^{23} \times a = 252.9 \text{ m}^2/\text{g}$. The assumed a and model control this estimate.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use the instructor's assigned material and apparatus; record actual temperature and units with every measured property.
- Check instrument calibration and use repeat readings rather than a single convenient result.
- Compare a marketed teaching formulation only under the same method and conditions; its excipients can change the property.
- Do not accept an adsorption curve without checking blank effects, equilibrium and appropriate model behaviour.

Viva Questions and Answers

1. Why distinguish this from BET surface area?

The experiment uses aqueous adsorption with model assumptions, not gas adsorption under BET conditions.

2. Why determine C_e rather than use C_0 ?

Adsorption is related to the concentration remaining at equilibrium.

3. What if the Langmuir fit is unsuitable?

Report measured adsorption and the failed model assumption rather than forcing a surface-area value.

Buffer Capacity and pKa During Weak-Acid Titration

Experiment 15 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To determine local buffer capacity and estimate pKa for a suitable weak-acid/strong-base system.

Principle

A weak acid and its conjugate base resist pH change most effectively around comparable concentrations. Buffer capacity is the amount of added strong acid/base per solution volume per pH change. For an ideal monoprotic weak acid, pH at half-equivalence approximates pKa.

Requirements

- Assigned weak-acid solution, standardised strong base and water control.
- Burette, calibrated pH meter, stirrer, volumetric glassware and temperature record.

Procedure

1. Calibrate

Calibrate the pH meter with appropriate buffers at the working temperature. Record acid identity, volume and concentration.

2. Titrate

Add measured base increments, allow mixing/equilibration and record pH and cumulative volume. Use smaller increments near important transitions.

3. Find equivalence

Determine equivalence from the titration curve using the assigned method rather than merely assuming an indicator colour.

4. Estimate pKa

Read pH at half the measured equivalence volume for a suitable monoprotic system. State limitations for concentrated, mixed or polyprotic systems.

5. Capacity

For small intervals calculate $\beta \approx \Delta n_{\text{base}} / (V_{\text{average}} \Delta \text{pH})$, using actual interval solution volume in litres. Compare early, half-equivalence and later stages.

6. Compare

Plot capacity against titration stage/pH and compare with the water control. Large intervals only give average capacity and can mask local behaviour.

Physical Pharmaceutics

Observations and Record

Cumulative base (mL)	pH / total solution volume	Interval β and units
_____	_____	_____
_____	_____	_____
_____	_____	_____

Worked Example

The numbers below are illustrative; enter your own observations in the record.

- If $\Delta n_{\text{base}} = 0.000200$ mol, average solution volume = 0.102 L and $\Delta \text{pH} = 0.200$, $\beta \approx 0.00980$ mol/L per pH unit.
- For 20.0 mL of 0.100 mol/L monoprotic acid titrated with 0.100 mol/L base, theoretical equivalence is 20.0 mL; half-equivalence is 10.0 mL.
- An illustrative half-equivalence pH of 4.76 gives $\text{pK}_a \approx 4.76$ under the stated approximation.

Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

Precautions

- Use the instructor's assigned material and apparatus; record actual temperature and units with every measured property.
- Check instrument calibration and use repeat readings rather than a single convenient result.
- Compare a marketed teaching formulation only under the same method and conditions; its excipients can change the property.
- Use actual titrant standardisation and account for added volume; rinse the electrode without wiping its sensitive surface harshly.

Viva Questions and Answers

1. Where is a simple buffer usually strongest?

Near $\text{pH} = \text{pK}_a$ when acid and conjugate-base concentrations are comparable.

2. Why include solution volume in capacity?

Capacity is expressed per litre, and titrant addition changes that volume.

3. Is half-equivalence pH always a precise thermodynamic pK_a ?

No. Activity, concentration and the chemical system affect the approximation.

Continue Learning with FirstHope

Complete Notes on the FirstHope App

Complete subject and semesters are available on FirstHope app only.

- Get complete subject and semester notes in the FirstHope app.
- Available for B.Pharm, D.Pharm, M.Pharm and Pharm.D students.

[Click here to get the FirstHope app](#)

Join the FirstHope Telegram Channel

Join FirstHope Notes and keep up with channel updates.

[Click here to join Telegram](#)

Instagram

@firsthope.official

[Click here to follow Instagram](#)

YouTube Channel

FirstHope-Pharmacy / Live streams

[Click here to watch the YouTube channel](#)

WhatsApp Channel

Follow FirstHope on WhatsApp.

[Click here to join the WhatsApp channel](#)

FirstHope Website

firsthopenotes.com

[Click here to visit the website](#)