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Semester 3 - Practical Notes

Physical Pharmaceutics I

Complete Subject Manual

11 practicals and activities. Use the PDF bookmarks to move between practicals. Page numbers restart within each practical; the page counts below exclude the promotion pages.

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Determination of Drug Solubility at Room Temperature

Practical 01 | Physical Pharmaceutics I

Aim

To determine Drug Solubility at Room Temperature, and interpret the recorded observations.

Principle

- Solubility is the equilibrium concentration of a solute in the specified solvent at a stated temperature.
- The property is obtained from a defined observable under controlled conditions.
- Replicate measurements, instrument checks and consistent units are needed before comparison.

Materials and Apparatus

- Assigned drug, solvent, equilibration vessel and the assigned concentration assay.
- Assigned test material and calibration or comparison material.
- The apparatus specified for this property, a balance or volumetric equipment where relevant, and a record sheet.

Method

1. Identify the drug form, solvent and measurement temperature.
2. Contact the assigned excess drug with the solvent using the instructor's equilibration arrangement.
3. Check the completed equilibration record and separate undissolved material without changing the solution concentration.
4. Measure a defined clear solution portion by the assigned concentration assay.
5. Correct for preparation/dilution and compare replicate equilibrium readings.
6. Report solubility with solvent, temperature and units.

Observation Record

Complete this record using actual laboratory findings or the provided teaching data.

Observation / parameter	Record / finding
Temperature	_____
Equilibration observations	_____
Aliquot dilution	_____
Measured concentration	_____
Replicates	_____

Calculations and Interpretation

- Solubility = measured equilibrium concentration $\times$ dilution factor.
- State concentration units, solvent and temperature.

Result

Record the actual finding, calculated value with units where relevant, and a conclusion supported by the observations.

Finding: _____

Conclusion: _____

Precautions

- Keep temperature and test conditions consistent across comparative readings.
- Record raw readings before rounding calculated values.
- Do not compare results obtained with different methods as if they were interchangeable.

Viva Questions

1. Why control the test conditions?

A change in conditions may alter the measured property independently of the intended variable.

2. Why retain replicate readings?

They show repeatability and help identify an anomalous measurement.

Determination of pKa by Half Neutralization

Practical 02 | Physical Pharmaceutics I

Aim

To determine pKa by Half Neutralization, and interpret the recorded observations.

Principle

- For a monoprotic weak acid, the Henderson-Hasselbalch relation gives $\text{pH} = \text{pKa}$ when the amounts of acid and conjugate base are equal.
- The property is obtained from a defined observable under controlled conditions.
- Replicate measurements, instrument checks and consistent units are needed before comparison.

Materials and Apparatus

- Assigned weak acid, standardized base, calibrated pH meter and titration glassware.
- Assigned test material and calibration or comparison material.
- The apparatus specified for this property, a balance or volumetric equipment where relevant, and a record sheet.

Method

1. Measure the assigned monoprotic weak-acid portion and calibrate the pH meter.
2. Add the standardized base incrementally and retain the volume-pH readings.
3. Identify the equivalence volume from the appropriate titration-curve interpretation.
4. Read the pH at half that equivalence volume.
5. Estimate pKa from the half-neutralization pH under the stated weak-acid assumptions.
6. State the temperature and explain possible effects of concentration or nonideal behaviour.

Observation Record

Complete this record using actual laboratory findings or the provided teaching data.

Observation / parameter	Record / finding
Added base volume	_____
pH	_____
Equivalence-volume estimate	_____
Half-equivalence pH	_____

Calculations and Interpretation

- $\text{pH} = \text{pK}_a + \log_{10}([\text{A}^-]/[\text{HA}])$.
- At half neutralization, $\text{pK}_a = \text{pH}$ for the stated weak-acid model.

Worked Illustration

Illustration: an equivalence volume of 20.0 mL makes the half-equivalence volume 10.0 mL. If its measured pH is 4.20 under the stated weak-acid model, pK_a is approximately 4.20.

The values in this illustration are for calculation practice.

Result

Record the actual finding, calculated value with units where relevant, and a conclusion supported by the observations.

Finding: _____

Conclusion: _____

Precautions

- Keep temperature and test conditions consistent across comparative readings.
- Record raw readings before rounding calculated values.
- Do not compare results obtained with different methods as if they were interchangeable.

Viva Questions

1. Why control the test conditions?

A change in conditions may alter the measured property independently of the intended variable.

2. Why retain replicate readings?

They show repeatability and help identify an anomalous measurement.

Determination of Benzoic Acid Partition Between Benzene and Water

Practical 03 | Physical Pharmaceutics I

Aim

To determine Benzoic Acid Partition Between Benzene and Water, and interpret the recorded observations.

Principle

- The measured total-concentration distribution ratio depends on pH and possible association in the organic phase. It is not automatically the intrinsic partition constant of one molecular species.
- The property is obtained from a defined observable under controlled conditions.
- Replicate measurements, instrument checks and consistent units are needed before comparison.

Materials and Apparatus

- Instructor-provided equilibrium phase solutions, assigned phase assay and closed partition apparatus.
- Assigned test material and calibration or comparison material.
- The apparatus specified for this property, a balance or volumetric equipment where relevant, and a record sheet.

Method

1. Identify the measured property, units and controlled variables before setting up the apparatus.
2. Check the instrument, zero or calibration and record the relevant operating conditions.
3. Prepare a representative measured portion without changing its intended composition.
4. Obtain the readings specified for the property and repeat under the same conditions.
5. Calculate the required property, including corrections stated for the apparatus or method.
6. Summarize the replicate results and explain the effect of the variable studied.

Observation Record

Complete this record using actual laboratory findings or the provided teaching data.

Observation / parameter	Record / finding
Phase volumes	_____
Temperature	_____
Aqueous pH	_____
Organic concentration	_____
Aqueous concentration	_____

Calculations and Interpretation

- Observed distribution ratio $D = \text{total concentration in organic phase} / \text{total concentration in aqueous phase}$.
- Keep units equal. Explain ionization or dimerization before treating D as an intrinsic constant.

Result

Record the actual finding, calculated value with units where relevant, and a conclusion supported by the observations.

Finding: _____

Conclusion: _____

Precautions

- Keep temperature and test conditions consistent across comparative readings.
- Record raw readings before rounding calculated values.
- Do not compare results obtained with different methods as if they were interchangeable.

Viva Questions

1. Why control the test conditions?

A change in conditions may alter the measured property independently of the intended variable.

2. Why retain replicate readings?

They show repeatability and help identify an anomalous measurement.

Determination of Iodine Partition Between Carbon Tetrachloride and Water

Practical 04 | Physical Pharmaceutics I

Aim

To determine Iodine Partition Between Carbon Tetrachloride and Water, and interpret the recorded observations.

Principle

- A distribution comparison uses equilibrium concentrations in two immiscible phases at the same temperature; added complexing solutes can alter the observed result.
- The property is obtained from a defined observable under controlled conditions.
- Replicate measurements, instrument checks and consistent units are needed before comparison.

Materials and Apparatus

- Instructor-provided equilibrium phase solutions and the assigned iodine assay.
- Assigned test material and calibration or comparison material.
- The apparatus specified for this property, a balance or volumetric equipment where relevant, and a record sheet.

Method

1. Identify the measured property, units and controlled variables before setting up the apparatus.
2. Check the instrument, zero or calibration and record the relevant operating conditions.
3. Prepare a representative measured portion without changing its intended composition.
4. Obtain the readings specified for the property and repeat under the same conditions.
5. Calculate the required property, including corrections stated for the apparatus or method.
6. Summarize the replicate results and explain the effect of the variable studied.

Observation Record

Complete this record using actual laboratory findings or the provided teaching data.

Observation / parameter	Record / finding
Phase identity	_____
Temperature	_____
Aliquot volumes	_____
Corrected assay readings	_____
Equilibrium concentrations	_____

Calculations and Interpretation

- Distribution ratio = iodine concentration in organic phase / iodine concentration in aqueous phase.
- Correct phase aliquots for dilution and keep the specified chemical composition unchanged.

Result

Record the actual finding, calculated value with units where relevant, and a conclusion supported by the observations.

Finding: _____

Conclusion: _____

Precautions

- Keep temperature and test conditions consistent across comparative readings.
- Record raw readings before rounding calculated values.
- Do not compare results obtained with different methods as if they were interchangeable.

Viva Questions

1. Why control the test conditions?

A change in conditions may alter the measured property independently of the intended variable.

2. Why retain replicate readings?

They show repeatability and help identify an anomalous measurement.

Determination of Sodium Chloride by Critical Solution Temperature

Practical 05 | Physical Pharmaceutics I

Aim

To determine Sodium Chloride by Critical Solution Temperature, and interpret the recorded observations.

Principle

- Salt changes the mutual-solubility behaviour of the phenol-water system. An empirical calibration relates the measured critical solution temperature to composition under specified conditions.
- The property is obtained from a defined observable under controlled conditions.
- Replicate measurements, instrument checks and consistent units are needed before comparison.

Materials and Apparatus

- Assigned phenol-water/salt demonstration, temperature measurement system and calibration records.
- Assigned test material and calibration or comparison material.
- The apparatus specified for this property, a balance or volumetric equipment where relevant, and a record sheet.

Method

1. Identify the measured property, units and controlled variables before setting up the apparatus.
2. Check the instrument, zero or calibration and record the relevant operating conditions.
3. Prepare a representative measured portion without changing its intended composition.
4. Obtain the readings specified for the property and repeat under the same conditions.
5. Calculate the required property, including corrections stated for the apparatus or method.
6. Summarize the replicate results and explain the effect of the variable studied.

Observation Record

Complete this record using actual laboratory findings or the provided teaching data.

Observation / parameter	Record / finding
Mixture composition	_____
Clearing/clouding readings	_____
Replicates	_____
Calibration concentration	_____

Calculations and Interpretation

- Estimate NaCl composition from the calibration relating composition to CST.
- Use only the composition and temperature range represented by that calibration.

Result

Record the actual finding, calculated value with units where relevant, and a conclusion supported by the observations.

Finding: _____

Conclusion: _____

Precautions

- Keep temperature and test conditions consistent across comparative readings.
- Record raw readings before rounding calculated values.
- Do not compare results obtained with different methods as if they were interchangeable.

Viva Questions

1. Why control the test conditions?

A change in conditions may alter the measured property independently of the intended variable.

2. Why retain replicate readings?

They show repeatability and help identify an anomalous measurement.

Determination of Surface Tension by Drop Count and Drop Weight

Practical 06 | Physical Pharmaceutics I

Aim

To determine Surface Tension by Drop Count and Drop Weight, and interpret the recorded observations.

Principle

- Drop methods compare liquid behaviour at the same capillary tip; density, temperature and drop-shape corrections affect the result.
- The property is obtained from a defined observable under controlled conditions.
- Replicate measurements, instrument checks and consistent units are needed before comparison.

Materials and Apparatus

- Stalagmometer or drop apparatus, comparison liquid, balance and density readings.
- Assigned test material and calibration or comparison material.
- The apparatus specified for this property, a balance or volumetric equipment where relevant, and a record sheet.

Method

1. Identify the measured property, units and controlled variables before setting up the apparatus.
2. Check the instrument, zero or calibration and record the relevant operating conditions.
3. Prepare a representative measured portion without changing its intended composition.
4. Obtain the readings specified for the property and repeat under the same conditions.
5. Calculate the required property, including corrections stated for the apparatus or method.
6. Summarize the replicate results and explain the effect of the variable studied.

Observation Record

Complete this record using actual laboratory findings or the provided teaching data.

Observation / parameter	Record / finding
Temperature	_____
Test/reference density	_____
Drop counts	_____
Mean drop masses	_____

Calculations and Interpretation

- Drop count: $\gamma_x/\gamma_r = (\rho_x \times n_r)/(\rho_r \times n_x)$ for the same counted volume.
- Drop weight: γ_x/γ_r approximately equals mean drop-mass ratio when the same tip and appropriate corrections apply.

Result

Record the actual finding, calculated value with units where relevant, and a conclusion supported by the observations.

Finding: _____

Conclusion: _____

Precautions

- Keep temperature and test conditions consistent across comparative readings.
- Record raw readings before rounding calculated values.
- Do not compare results obtained with different methods as if they were interchangeable.

Viva Questions

1. Why control the test conditions?

A change in conditions may alter the measured property independently of the intended variable.

2. Why retain replicate readings?

They show repeatability and help identify an anomalous measurement.

Determination of HLB by Saponification Method

Practical 07 | Physical Pharmaceutics I

Aim

To determine HLB by Saponification Method, and interpret the recorded observations.

Principle

- For appropriate ester-type nonionic surfactants, the Griffin saponification relation estimates hydrophilic-lipophilic balance from saponification and fatty-acid acid values.
- The property is obtained from a defined observable under controlled conditions.
- Replicate measurements, instrument checks and consistent units are needed before comparison.

Materials and Apparatus

- Assigned ester surfactant, saponification data and acid-value data for the constituent fatty acid.
- Assigned test material and calibration or comparison material.
- The apparatus specified for this property, a balance or volumetric equipment where relevant, and a record sheet.

Method

1. Identify the measured property, units and controlled variables before setting up the apparatus.
2. Check the instrument, zero or calibration and record the relevant operating conditions.
3. Prepare a representative measured portion without changing its intended composition.
4. Obtain the readings specified for the property and repeat under the same conditions.
5. Calculate the required property, including corrections stated for the apparatus or method.
6. Summarize the replicate results and explain the effect of the variable studied.

Observation Record

Complete this record using actual laboratory findings or the provided teaching data.

Observation / parameter	Record / finding
Surfactant identity	_____
Saponification value S	_____
Acid value A	_____
Calculated HLB	_____

Calculations and Interpretation

- $HLB = 20(1 - S/A)$.
- Apply this empirical expression only to the surfactant class and acid-value basis for which it is valid.

Result

Record the actual finding, calculated value with units where relevant, and a conclusion supported by the observations.

Finding: _____

Conclusion: _____

Precautions

- Keep temperature and test conditions consistent across comparative readings.
- Record raw readings before rounding calculated values.
- Do not compare results obtained with different methods as if they were interchangeable.

Viva Questions

1. Why control the test conditions?

A change in conditions may alter the measured property independently of the intended variable.

2. Why retain replicate readings?

They show repeatability and help identify an anomalous measurement.

Determination of Freundlich and Langmuir Adsorption Constants

Practical 08 | Physical Pharmaceutics I

Aim

To determine Freundlich and Langmuir Adsorption Constants, and interpret the recorded observations.

Principle

- Adsorption is calculated from the loss of dissolved solute after contact with a known adsorbent mass. Freundlich is empirical; Langmuir describes a stated idealized adsorption model.
- The property is obtained from a defined observable under controlled conditions.
- Replicate measurements, instrument checks and consistent units are needed before comparison.

Materials and Apparatus

- Activated charcoal, assigned adsorbate solutions and concentration-assay apparatus.
- Assigned test material and calibration or comparison material.
- The apparatus specified for this property, a balance or volumetric equipment where relevant, and a record sheet.

Method

1. Identify the measured property, units and controlled variables before setting up the apparatus.
2. Check the instrument, zero or calibration and record the relevant operating conditions.
3. Prepare a representative measured portion without changing its intended composition.
4. Obtain the readings specified for the property and repeat under the same conditions.
5. Calculate the required property, including corrections stated for the apparatus or method.
6. Summarize the replicate results and explain the effect of the variable studied.

Observation Record

Complete this record using actual laboratory findings or the provided teaching data.

Observation / parameter	Record / finding
Initial concentration C_0	_____
Equilibrium concentration C_e	_____
Solution volume V	_____
Charcoal mass m	_____
q_e	_____

Calculations and Interpretation

- $q_e = (C_0 - C_e)V/m$, with consistent concentration/volume units.
- Freundlich: $\log(q_e) = \log(KF) + (1/n)\log(C_e)$.
- Langmuir: $C_e/q_e = 1/(q_{\max} KL) + C_e/q_{\max}$.
- A fitted line alone does not prove the physical adsorption mechanism.

Result

Record the actual finding, calculated value with units where relevant, and a conclusion supported by the observations.

Finding: _____

Conclusion: _____

Precautions

- Keep temperature and test conditions consistent across comparative readings.
- Record raw readings before rounding calculated values.
- Do not compare results obtained with different methods as if they were interchangeable.

Viva Questions

1. Why control the test conditions?

A change in conditions may alter the measured property independently of the intended variable.

2. Why retain replicate readings?

They show repeatability and help identify an anomalous measurement.

Determination of Critical Micelle Concentration

Practical 09 | Physical Pharmaceutics I

Aim

To determine Critical Micelle Concentration, and interpret the recorded observations.

Principle

- A change in a concentration-dependent property identifies the transition associated with micelle formation. The breakpoint is method- and condition-dependent.
- The property is obtained from a defined observable under controlled conditions.
- Replicate measurements, instrument checks and consistent units are needed before comparison.

Materials and Apparatus

- Assigned surfactant series and conductivity or surface-tension apparatus.
- Assigned test material and calibration or comparison material.
- The apparatus specified for this property, a balance or volumetric equipment where relevant, and a record sheet.

Method

1. Identify the measured property, units and controlled variables before setting up the apparatus.
2. Check the instrument, zero or calibration and record the relevant operating conditions.
3. Prepare a representative measured portion without changing its intended composition.
4. Obtain the readings specified for the property and repeat under the same conditions.
5. Calculate the required property, including corrections stated for the apparatus or method.
6. Summarize the replicate results and explain the effect of the variable studied.

Observation Record

Complete this record using actual laboratory findings or the provided teaching data.

Observation / parameter	Record / finding
Surfactant concentration	_____
Temperature	_____
Measured property	_____
Breakpoint estimate	_____

Calculations and Interpretation

- Plot the chosen property against concentration or log concentration as appropriate.
- Estimate CMC from the intersection of the two fitted regions and state the units.

Result

Record the actual finding, calculated value with units where relevant, and a conclusion supported by the observations.

Finding: _____

Conclusion: _____

Precautions

- Keep temperature and test conditions consistent across comparative readings.
- Record raw readings before rounding calculated values.
- Do not compare results obtained with different methods as if they were interchangeable.

Viva Questions

1. Why control the test conditions?

A change in conditions may alter the measured property independently of the intended variable.

2. Why retain replicate readings?

They show repeatability and help identify an anomalous measurement.

Determination of PABA-Caffeine Complex by Solubility Method

Practical 10 | Physical Pharmaceutics I

Aim

To determine PABA-Caffeine Complex by Solubility Method, and interpret the recorded observations.

Principle

- A phase-solubility plot compares drug solubility across ligand concentration. A simple AL plot can support a conditional 1:1 association model.
- The property is obtained from a defined observable under controlled conditions.
- Replicate measurements, instrument checks and consistent units are needed before comparison.

Materials and Apparatus

- Assigned PABA/caffeine phase-solubility data or supervised solutions and concentration assay.
- Assigned test material and calibration or comparison material.
- The apparatus specified for this property, a balance or volumetric equipment where relevant, and a record sheet.

Method

1. Identify the measured property, units and controlled variables before setting up the apparatus.
2. Check the instrument, zero or calibration and record the relevant operating conditions.
3. Prepare a representative measured portion without changing its intended composition.
4. Obtain the readings specified for the property and repeat under the same conditions.
5. Calculate the required property, including corrections stated for the apparatus or method.
6. Summarize the replicate results and explain the effect of the variable studied.

Observation Record

Complete this record using actual laboratory findings or the provided teaching data.

Observation / parameter	Record / finding
Ligand concentration	_____
Total dissolved PABA	_____
Intrinsic solubility S_0	_____
Plot slope	_____

Calculations and Interpretation

- For a valid 1:1 AL model: $K = \text{slope} / [S_0(1 - \text{slope})]$.
- Use molar units, slope below one and the stated pH/temperature; do not apply the expression to every plot shape.

Result

Record the actual finding, calculated value with units where relevant, and a conclusion supported by the observations.

Finding: _____

Conclusion: _____

Precautions

- Keep temperature and test conditions consistent across comparative readings.
- Record raw readings before rounding calculated values.
- Do not compare results obtained with different methods as if they were interchangeable.

Viva Questions

1. Why control the test conditions?

A change in conditions may alter the measured property independently of the intended variable.

2. Why retain replicate readings?

They show repeatability and help identify an anomalous measurement.

Determination of Cupric-Glycine Complex by pH Titration

Practical 11 | Physical Pharmaceutics I

Aim

To determine Cupric-Glycine Complex by pH Titration, and interpret the recorded observations.

Principle

- Ligand binding can alter proton release and the titration curve. Comparison of metal-free and metal-containing systems helps interpret conditional complex formation.
- The property is obtained from a defined observable under controlled conditions.
- Replicate measurements, instrument checks and consistent units are needed before comparison.

Materials and Apparatus

- Assigned glycine/copper titration records, pH meter and control titration.
- Assigned test material and calibration or comparison material.
- The apparatus specified for this property, a balance or volumetric equipment where relevant, and a record sheet.

Method

1. Identify the measured property, units and controlled variables before setting up the apparatus.
2. Check the instrument, zero or calibration and record the relevant operating conditions.
3. Prepare a representative measured portion without changing its intended composition.
4. Obtain the readings specified for the property and repeat under the same conditions.
5. Calculate the required property, including corrections stated for the apparatus or method.
6. Summarize the replicate results and explain the effect of the variable studied.

Observation Record

Complete this record using actual laboratory findings or the provided teaching data.

Observation / parameter	Record / finding
Added titrant	_____
pH of ligand control	_____
pH of metal-ligand system	_____
Assigned stoichiometry	_____

Calculations and Interpretation

- For an assumed ML complex, conditional $K = [ML]/([M]_{\text{free}}[L]_{\text{free}})$.
- Establish the relevant stoichiometry and free-species estimates before substituting values.

Result

Record the actual finding, calculated value with units where relevant, and a conclusion supported by the observations.

Finding: _____

Conclusion: _____

Precautions

- Keep temperature and test conditions consistent across comparative readings.
- Record raw readings before rounding calculated values.
- Do not compare results obtained with different methods as if they were interchangeable.

Viva Questions

1. Why control the test conditions?

A change in conditions may alter the measured property independently of the intended variable.

2. Why retain replicate readings?

They show repeatability and help identify an anomalous measurement.

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