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Semester 4 - Practical Notes

Physical Pharmaceutics II

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.

Contents

No.	Practical / Activity	Pages
1	Determination of Particle Size by Sieving	2
2	Determination of Particle Size by Microscopy	2
3	Determination of Bulk Density, True Density and Porosity	2
4	Determination of Angle of Repose and Lubricant Effect	2
5	Determination of Liquid Viscosity by Ostwald Viscometer	2
6	Determination of Sedimentation Volume with Different Suspending Agents	2
7	Determination of Sedimentation Volume at Different Agent Concentrations	2
8	Determination of Semisolid Viscosity by Brookfield Viscometer	2
9	Determination of First-Order Reaction Rate Constant	2
10	Determination of Second-Order Reaction Rate Constant	2
11	Determination of Accelerated Stability Studies	2

Determination of Particle Size by Sieving

Practical 01 | Physical Pharmaceutics II

Aim

To determine Particle Size by Sieving, and interpret the recorded observations.

Principle

- A sieve stack divides a powder by aperture-related fractions and provides a mass-weighted size distribution.
- 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

- Sieve stack, sieve shaker, balance and assigned powder.
- 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. Weigh the assigned powder and record the sieve apertures.
2. Arrange the stack in the stated descending-aperture order with a receiving pan.
3. Run the assigned sieving demonstration and record its duration and conditions.
4. Weigh each retained fraction and check total recovered mass.
5. Calculate percentage retained and cumulative retained or passing values.
6. Plot the size-distribution result with its mass-weighted basis stated.

Observation Record

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

Observation / parameter	Record / finding
Sieve aperture	_____
Retained mass	_____
Percent retained	_____
Cumulative passing	_____

Calculations and Interpretation

- Percent retained = retained mass/total recovered mass × 100.
- Use the stated fraction-size convention when calculating a mean size.

Worked Illustration

Illustration: a fraction of 8.0 g from 40.0 g total recovered powder represents 20.0% retained.

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 Particle Size by Microscopy

Practical 02 | Physical Pharmaceutics II

Aim

To determine Particle Size by Microscopy, and interpret the recorded observations.

Principle

- Microscopy measures selected projected dimensions of individual particles. The resulting number-weighted distribution differs from a mass-weighted sieve distribution.
- 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

- Prepared particle mounts, microscope and calibrated micrometer.
- 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. Calibrate the ocular scale against the stage micrometer for the selected objective.
2. Observe representative fields of the prepared particle mount.
3. Define the dimension being measured and measure particles without selecting only large or clear examples.
4. Convert ocular divisions to micrometres with the correct calibration.
5. Group sizes into the assigned intervals and calculate the number-weighted distribution.
6. Report the dimension definition, counting basis and distribution summary.

Observation Record

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

Observation / parameter	Record / finding
Calibration	_____
Particle dimension	_____
Count per size band	_____
Mean size	_____

Calculations and Interpretation

- $\text{Size} = \text{ocular divisions} \times \text{calibration (micrometres/division)}$.
- $\text{Number mean size} = \frac{\sum(n_i \times d_i)}{\sum(n_i)}$.

Worked Illustration

Illustration: calibration of 2.5 micrometres/division and an object length of 8 divisions give 20 micrometres.

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 Bulk Density, True Density and Porosity

Practical 03 | Physical Pharmaceutics II

Aim

To determine Bulk Density, True Density and Porosity, and interpret the recorded observations.

Principle

- Bulk density includes interparticle void space; true density refers to the material itself under the stated measurement method.
- 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 powder, graduated cylinder and approved true-density method or 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
Powder mass	_____
Bulk volume	_____
True volume/density	_____
Porosity	_____

Calculations and Interpretation

- Bulk density = mass/bulk volume.
- True density = mass/true material volume.
- Interparticle porosity = $1 - \text{bulk density/true density}$; multiply by 100 for percentage.

Worked Illustration

Illustration: 25.0 g in a 50.0 mL bulk volume has bulk density 0.500 g/mL. If true density is 1.25 g/mL, calculated interparticle porosity is 60.0%.

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 Angle of Repose and Lubricant Effect

Practical 04 | Physical Pharmaceutics II

Aim

To determine Angle of Repose and Lubricant Effect, and interpret the recorded observations.

Principle

- The angle formed by a powder heap is a method-dependent indicator of flow behaviour. Lubricant identity and concentration can change interparticle interactions.
- 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 powder/lubricant comparisons and fixed-height or assigned cone 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. Record the powder condition and lubricant identity/concentration.
2. Set the assigned funnel/cone apparatus without changing its geometry between comparisons.
3. Form the heap by the specified procedure and measure height and base radius.
4. Repeat with a fresh representative portion.
5. Calculate the angle from $\arctan(\text{height/radius})$.
6. Compare preparations and explain why the method and powder history affect the result.

Observation Record

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

Observation / parameter	Record / finding
Powder/lubricant condition	_____
Cone height h	_____
Cone radius r	_____
Replicates	_____

Calculations and Interpretation

- $\tan(\theta) = h/r$; $\theta = \arctan(h/r)$.
- Compare preparations using the same cone-building method.

Worked Illustration

Illustration: a heap 3.0 cm high with 5.0 cm radius gives $\theta = \arctan(3/5) = 31.0$ degrees.

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 Liquid Viscosity by Ostwald Viscometer

Practical 05 | Physical Pharmaceutics II

Aim

To determine Liquid Viscosity by Ostwald Viscometer, and interpret the recorded observations.

Principle

- For suitable Newtonian liquids in the same viscometer, flow time and density support a viscosity comparison at constant 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

- Ostwald viscometer, reference liquid, test liquid, timer and temperature control.
- 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. Check that the assigned liquid is suitable for the comparison method and record temperature.
2. Measure the densities of the reference and test liquids or use the approved values.
3. Measure reference-liquid flow time between the stated marks.
4. Measure test-liquid flow time in the same viscometer at the same temperature.
5. Repeat readings and calculate the viscosity ratio from density and time.
6. Report viscosity with the reference value and controlled temperature.

Observation Record

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

Observation / parameter	Record / finding
Temperature	_____
Reference/test density	_____
Reference/test flow time	_____

Calculations and Interpretation

- $\eta_{\text{test}}/\eta_{\text{reference}} = (\rho_{\text{test}} \times t_{\text{test}})/(\rho_{\text{reference}} \times t_{\text{reference}})$.
- State the reference viscosity and temperature.

Worked Illustration

Illustration: $\eta_{\text{reference}} = 1.00 \text{ mPa s}$, $\rho_{\text{test}}/\rho_{\text{reference}} = 1.10$ and $t_{\text{test}}/t_{\text{reference}} = 1.50$ give $\eta_{\text{test}} = 1.65 \text{ mPa s}$.

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 Sedimentation Volume with Different Suspending Agents

Practical 06 | Physical Pharmaceutics II

Aim

To determine Sedimentation Volume with Different Suspending Agents, and interpret the recorded observations.

Principle

- The ultimate sediment volume is compared with initial suspension volume. A large sedimentation volume does not alone establish easy redispersion or product quality.
- 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

- Comparable suspensions containing different assigned suspending agents and graduated containers.
- 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. Prepare or identify the comparable assigned suspensions and record their initial volume.
2. Document the suspending-agent identity and concentration for every container.
3. Record sediment volume at the specified observation times without disturbing the sediment prematurely.
4. Assess redispersion by the assigned procedure and record it separately.
5. Calculate the volume ratio and compare at the same time point.
6. Discuss settling, sediment structure and redispersion together.

Observation Record

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

Observation / parameter	Record / finding
Agent	_____
Initial volume V_0	_____
Sediment volume V_u	_____
Time	_____
Redispersibility	_____

Calculations and Interpretation

- Sedimentation volume $F = V_u/V_0$.
- Compare like-for-like sediment readings and include redispersion observations.

Worked Illustration

Illustration: 18.0 mL final sediment in a suspension initially occupying 30.0 mL gives $F = 0.60$. Redispersion still needs separate observation.

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 Sedimentation Volume at Different Agent Concentrations

Practical 07 | Physical Pharmaceutics II

Aim

To determine Sedimentation Volume at Different Agent Concentrations, and interpret the recorded observations.

Principle

- Changing the concentration of one suspending agent can alter viscosity, particle settling and sediment structure.
- 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

- Same drug dispersion and graded concentrations of one assigned suspending agent.
- 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. Prepare or identify the comparable assigned suspensions and record their initial volume.
2. Document the suspending-agent identity and concentration for every container.
3. Record sediment volume at the specified observation times without disturbing the sediment prematurely.
4. Assess redispersion by the assigned procedure and record it separately.
5. Calculate the volume ratio and compare at the same time point.
6. Discuss settling, sediment structure and redispersion together.

Observation Record

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

Observation / parameter	Record / finding
Agent concentration	_____
Time	_____
V ₀	_____
V _u	_____
Redispersion observations	_____

Calculations and Interpretation

- $F = V_u/V_0$.
- Plot F against the agent concentration at the same observation time.

Worked Illustration

Illustration: 18.0 mL final sediment in a suspension initially occupying 30.0 mL gives $F = 0.60$. Redispersion still needs separate observation.

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 Semisolid Viscosity by Brookfield Viscometer

Practical 08 | Physical Pharmaceutics II

Aim

To determine Semisolid Viscosity by Brookfield Viscometer, and interpret the recorded observations.

Principle

- A rotational viscometer reports an apparent viscosity linked to spindle, speed, temperature and measurement history. Non-Newtonian semisolids need these conditions stated.
- 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 semisolid, Brookfield-type viscometer and specified spindle.
- 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. Record the semisolid preparation and measurement temperature.
2. Select the assigned spindle and speed and check the instrument's valid torque range.
3. Position the spindle as specified and avoid entrained bubbles.
4. Obtain repeatable readings after the defined measurement history.
5. Record apparent viscosity together with spindle, speed, temperature and torque.
6. Compare readings only under stated equivalent conditions or explain the shear dependence.

Observation Record

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

Observation / parameter	Record / finding
Spindle	_____
Speed	_____
Temperature	_____
Torque validity	_____
Apparent viscosity	_____

Calculations and Interpretation

- Apply the instrument-approved spindle/speed factor or displayed result.
- Report apparent viscosity with spindle, speed and temperature; it is not necessarily one universal material 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 First-Order Reaction Rate Constant

Practical 09 | Physical Pharmaceutics II

Aim

To determine First-Order Reaction Rate Constant, and interpret the recorded observations.

Principle

- For a first-order process, the concentration declines exponentially and a logarithmic concentration-time plot is linear within the model range.
- 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 time-concentration data or supervised reaction-assay 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 concentration and time units in the assigned data.
2. Tabulate concentration with time and check the initial concentration.
3. Calculate natural logarithms and plot $\ln(\text{concentration})$ against time.
4. Fit the suitable model interval and record the slope.
5. Obtain k as the negative slope and calculate the half-life.
6. Inspect departures from linearity before accepting the first-order model.

Observation Record

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

Observation / parameter	Record / finding
Time t	_____
Initial concentration C ₀	_____
Concentration C _t	_____
ln(C _t)	_____
Fitted slope	_____

Calculations and Interpretation

- $\ln(C_t) = \ln(C_0) - kt$.
- $k = \ln(C_0/C_t)/t$; fitted $\ln(C_t)$ -versus-time slope = -k.
- Half-life = $0.693/k$ for the first-order model.

Worked Illustration

Illustration: C₀ = 10.0 mg/L and C_t = 5.00 mg/L after 2.00 hours give $k = \ln(2)/2 = 0.347$ per hour and half-life = 2.00 hours.

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 Second-Order Reaction Rate Constant

Practical 10 | Physical Pharmaceutics II

Aim

To determine Second-Order Reaction Rate Constant, and interpret the recorded observations.

Principle

- For the stated single-reactant second-order model, reciprocal concentration increases linearly with time. Other second-order stoichiometries need their own integrated equation.
- 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 concentration-time data and calculation software.
- 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 whether the supplied data fit the stated single-reactant second-order model.
2. Tabulate time, concentration and reciprocal concentration.
3. Plot reciprocal concentration against time.
4. Fit the appropriate interval and obtain k from its slope.
5. Calculate half-life with the initial concentration and retained units.
6. State the model rather than transferring the equation to a different stoichiometry.

Observation Record

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

Observation / parameter	Record / finding
Time t	_____
C ₀	_____
C _t	_____
1/C _t	_____
Fitted slope	_____

Calculations and Interpretation

- $1/C_t = 1/C_0 + kt$.
- $k = (1/C_t - 1/C_0)/t$ for this model.
- Half-life = $1/(kC_0)$.

Worked Illustration

Illustration for the stated model: $C_0 = 1.00$ mol/L, $C_t = 0.500$ mol/L at 10.0 min give $k = 0.100$ L/(mol min) and half-life = 10.0 min.

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 Accelerated Stability Studies

Practical 11 | Physical Pharmaceutics II

Aim

To determine Accelerated Stability Studies, and interpret the recorded observations.

Principle

- Temperature-dependent rate data can be examined with the Arrhenius model when the degradation mechanism and physical state remain comparable.
- 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 stability time/concentration records at several temperatures.
- 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. Review concentration-time records at the assigned temperatures.
2. Use the appropriate kinetic model to estimate a rate constant at each temperature.
3. Convert all temperatures to kelvin.
4. Plot $\ln(\text{rate constant})$ against reciprocal absolute temperature.
5. Interpret the fitted slope and any extrapolated rate only if the mechanism remains comparable.
6. Report model assumptions and distinguish a classroom estimate from a justified product shelf life.

Observation Record

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

Observation / parameter	Record / finding
Temperature in kelvin	_____
Concentration-time readings	_____
Rate constant	_____
Arrhenius plot	_____

Calculations and Interpretation

- $\ln(k) = \ln(A) - E_a/(RT)$.
- Slope of $\ln(k)$ versus $1/T = -E_a/R$.
- For first-order loss, $t_{90} = -\ln(0.90)/k$; extrapolation is conditional and does not itself authorize a shelf life.

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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