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Semester 7 - Practical Notes

Instrumental Methods of Analysis and Novel Drug Delivery Systems

Complete Subject Manual

18 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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Absorption Maxima and Solvent Effects

Practical 01 | Instrumental Methods of Analysis and Novel Drug Delivery Systems

Aim

To study Absorption Maxima and Solvent Effects, and interpret the recorded observations.

Principle

- An absorption maximum is the wavelength of a peak in the stated spectrum. Solvent polarity, hydrogen bonding, pH and analyte ionization can change the spectrum; their effects depend on the transition and molecular form.
- An optical response is related to analyte concentration over the validated working range.
- A blank, suitable standards and consistent optical conditions are necessary for interpreting the response.

Materials and Apparatus

- Assigned organic compound, appropriate solvents, UV-visible instrument and compatible cells.
- Assigned analyte, suitable reference standard, solvent/reagents and volumetric glassware.
- Specified optical instrument and compatible cells or sample-introduction system.

Method

1. Identify the analyte, solvent and relevant ionization conditions.
2. Prepare comparable solutions within a suitable optical range.
3. Run the solvent blank and record the spectrum for each solution.
4. Identify the peak wavelength without confusing it with an instrument artefact.
5. Compare spectral position and shape at documented concentration and conditions.
6. Explain any bathochromic or hypsochromic shift using the stated molecular/solvent context.

Observation Record

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

Observation / parameter	Record / finding
Compound/solvent	_____
pH where relevant	_____
Scan range	_____
Peak wavelength	_____
Peak absorbance	_____

Calculations and Interpretation

- Wavelength shift = test-solvent peak wavelength - comparison-solvent peak wavelength.
- Positive shift means movement to a longer wavelength; a shift direction cannot be assumed from polarity alone.

Worked Illustration

Illustration: a peak moving from 260 nm to 270 nm has a +10 nm wavelength shift, described as bathochromic.

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

- Use clean compatible cells and consistent orientation where cells are used.
- Keep readings within the calibration range and examine the calibration fit.
- Avoid assuming that another analyte or solvent has the same response factor.

Viva Questions

1. What is a bathochromic shift?

A movement of an absorption feature toward a longer wavelength.

2. What is the role of a blank?

It corrects the contribution of the solvent, reagents and measurement background.

3. Why use several calibration levels?

They establish the usable response-concentration relationship and help reveal nonlinearity.

Estimation of Sulphanilamide by Colorimetry

Practical 02 | Instrumental Methods of Analysis and Novel Drug Delivery Systems

Aim

To study Estimation of Sulphanilamide by Colorimetry, and interpret the recorded observations.

Principle

- The assigned colour reaction converts the analyte response into a visible-wavelength measurement. Standards, reagent blank and a controlled colour-development interval establish the calibration.
- An optical response is related to analyte concentration over the validated working range.
- A blank, suitable standards and consistent optical conditions are necessary for interpreting the response.
- Record the colour-reaction sequence demonstrated for this analyte and the wavelength selected.
- Prepare standards and test through the same reaction sequence so reagent and timing effects are comparable.

Materials and Apparatus

- Sulphanilamide standard, assigned colour-reaction system, colorimeter and volumetric apparatus.
- Assigned analyte, suitable reference standard, solvent/reagents and volumetric glassware.
- Specified optical instrument and compatible cells or sample-introduction system.

Method

1. Select the optical method and settings appropriate to the assigned analyte.
2. Prepare a method blank and a series of independently documented calibration concentrations.
3. Check the instrument response and measure the blank and standards using consistent settings.
4. Prepare the test solution and apply documented dilution where necessary.
5. Read the test response within the established calibration range.
6. Calculate concentration, correct for dilution and report the original-material basis.

Observation Record

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

Observation / parameter	Record / finding
Standard concentration	_____
Blank/standard response	_____
Colour-development interval	_____
Test absorbance	_____
Dilution	_____

Calculations and Interpretation

- For calibration response $y = mC + b$: $C = (y - b) / m$.
- Original concentration = concentration read from calibration $\times$ total dilution factor.

Worked Illustration

Illustration: $y = 0.050C + 0.010$ and $y = 0.260$ give $C = 5.00$ in the concentration units used for calibration.

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

- Use clean compatible cells and consistent orientation where cells are used.
- Keep readings within the calibration range and examine the calibration fit.
- Avoid assuming that another analyte or solvent has the same response factor.

Viva Questions

1. What is the role of a blank?

It corrects the contribution of the solvent, reagents and measurement background.

2. Why use several calibration levels?

They establish the usable response-concentration relationship and help reveal nonlinearity.

Simultaneous UV Estimation of Ibuprofen and Paracetamol

Practical 03 | Instrumental Methods of Analysis and Novel Drug Delivery Systems

Aim

To study Simultaneous UV Estimation of Ibuprofen and Paracetamol, and interpret the recorded observations.

Principle

- At two selected wavelengths, the total absorbance of a suitable two-component mixture can be represented as the sum of its component contributions. The absorptivity matrix must be nonsingular and the Beer-Lambert model valid in the chosen range.
- An optical response is related to analyte concentration over the validated working range.
- A blank, suitable standards and consistent optical conditions are necessary for interpreting the response.
- If the two components have nearly proportional responses at the chosen wavelengths, small absorbance errors can create large concentration errors.

Materials and Apparatus

- Pure component standards, assigned mixture, solvent and UV spectrophotometer.
- Assigned analyte, suitable reference standard, solvent/reagents and volumetric glassware.
- Specified optical instrument and compatible cells or sample-introduction system.

Method

1. Prepare separate component standards using the assigned solvent and reporting units.
2. Determine the component absorptivities at both selected wavelengths.
3. Measure the mixture absorbance at the same wavelengths and path length.
4. Check that the two-equation system is suitable and the response lies within the calibration range.
5. Solve for both concentrations and apply the actual dilution factor.
6. Report each component separately and assess the result against the prepared/declared mixture basis.

Observation Record

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

Observation / parameter	Record / finding
Wavelengths	_____
Component absorptivities	_____
Mixture absorbances	_____
Calculated concentrations	_____
Dilution	_____

Calculations and Interpretation

- $A_1 = a_{X1} CX + a_{Y1} CY$; $A_2 = a_{X2} CX + a_{Y2} CY$, for the same stated path length and units.
- $D = a_{X1} a_{Y2} - a_{X2} a_{Y1}$; D must be nonzero and sufficiently well conditioned.
- $CX = (A_1 a_{Y2} - A_2 a_{Y1})/D$.
- $CY = (a_{X1} A_2 - a_{X2} A_1)/D$.

Worked Illustration

Illustration: $A_1 = 0.300$, $A_2 = 0.350$ and $(a_{X1}, a_{Y1}, a_{X2}, a_{Y2}) = (0.020, 0.010, 0.005, 0.030)$ L/mg give $CX = CY = 10.0$ mg/L in the stated two-equation model.

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

- Use clean compatible cells and consistent orientation where cells are used.
- Keep readings within the calibration range and examine the calibration fit.
- Avoid assuming that another analyte or solvent has the same response factor.

Viva Questions

1. Why measure the pure components?

Their wavelength-specific responses supply the coefficients needed to separate the mixture contributions.

2. Why choose two suitably different wavelength responses?

Nearly proportional component responses make the equations poorly conditioned, so small measurement errors can produce large concentration errors.

Estimation of Quinine Sulphate by Fluorimetry

Practical 04 | Instrumental Methods of Analysis and Novel Drug Delivery Systems

Aim

To study Estimation of Quinine Sulphate by Fluorimetry, and interpret the recorded observations.

Principle

- Quinine fluorescence is measured at defined excitation/emission settings and solution conditions. Fluorescence is not interchangeable with absorbance, and pH or matrix effects can change the response.
- An optical response is related to analyte concentration over the validated working range.
- A blank, suitable standards and consistent optical conditions are necessary for interpreting the response.
- Keep the solvent, pH, instrument settings and cell arrangement consistent between standards and test.
- Use the working range demonstrated by the calibration; concentrated solutions can show inner-filter or other nonlinear effects.

Materials and Apparatus

- Quinine sulphate standard, assigned solvent, fluorimeter and suitable cells.
- Assigned analyte, suitable reference standard, solvent/reagents and volumetric glassware.
- Specified optical instrument and compatible cells or sample-introduction system.

Method

1. Select the optical method and settings appropriate to the assigned analyte.
2. Prepare a method blank and a series of independently documented calibration concentrations.
3. Check the instrument response and measure the blank and standards using consistent settings.
4. Prepare the test solution and apply documented dilution where necessary.
5. Read the test response within the established calibration range.
6. Calculate concentration, correct for dilution and report the original-material basis.

Observation Record

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

Observation / parameter	Record / finding
Standard concentration	_____
Excitation/emission settings	_____
Blank fluorescence	_____
Test fluorescence	_____
Dilution	_____

Calculations and Interpretation

- For calibration response $y = mC + b$: $C = (y - b) / m$.
- Original concentration = concentration read from calibration $\times$ total dilution factor.

Worked Illustration

Illustration: $y = 0.050C + 0.010$ and $y = 0.260$ give $C = 5.00$ in the concentration units used for calibration.

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

- Use clean compatible cells and consistent orientation where cells are used.
- Keep readings within the calibration range and examine the calibration fit.
- Avoid assuming that another analyte or solvent has the same response factor.

Viva Questions

1. What is the role of a blank?

It corrects the contribution of the solvent, reagents and measurement background.

2. Why use several calibration levels?

They establish the usable response-concentration relationship and help reveal nonlinearity.

Fluorescence Quenching

Practical 05 | Instrumental Methods of Analysis and Novel Drug Delivery Systems

Aim

To study Fluorescence Quenching, and interpret the recorded observations.

Principle

- A quencher can reduce fluorescence through different mechanisms. A Stern-Volmer relationship is appropriate for the stated simple dynamic-quenching model; static association and optical inner-filter effects require separate interpretation.
- An optical response is related to analyte concentration over the validated working range.
- A blank, suitable standards and consistent optical conditions are necessary for interpreting the response.

Materials and Apparatus

- Prepared fluorescent standard and quencher series, fluorimeter and completed comparative records.
- Assigned analyte, suitable reference standard, solvent/reagents and volumetric glassware.
- Specified optical instrument and compatible cells or sample-introduction system.

Method

1. Select the optical method and settings appropriate to the assigned analyte.
2. Prepare a method blank and a series of independently documented calibration concentrations.
3. Check the instrument response and measure the blank and standards using consistent settings.
4. Prepare the test solution and apply documented dilution where necessary.
5. Read the test response within the established calibration range.
6. Calculate concentration, correct for dilution and report the original-material basis.

Observation Record

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

Observation / parameter	Record / finding
Quencher concentration	_____
Unquenched F ₀	_____
Observed F	_____
F ₀ /F	_____
Model fit	_____

Calculations and Interpretation

- Simple dynamic Stern-Volmer model: $F_0/F = 1 + K_{SV}[Q]$.
- Plot F_0/F against quencher concentration; the slope is K_{SV} within the valid model.
- State concentration units because they determine the units of K_{SV} .

Worked Illustration

Illustration: $F_0 = 100$ and $F = 50$ give $F_0/F = 2.0$. Under the simple dynamic model at $[Q] = 0.010$ mol/L, $K_{SV} = 100$ L/mol.

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

- Use clean compatible cells and consistent orientation where cells are used.
- Keep readings within the calibration range and examine the calibration fit.
- Avoid assuming that another analyte or solvent has the same response factor.

Viva Questions

1. Does reduced intensity alone prove dynamic quenching?

No. Static association, absorption of excitation/emission light and instrumental effects can also reduce the observed signal.

2. What is the role of a blank?

It corrects the contribution of the solvent, reagents and measurement background.

3. Why use several calibration levels?

They establish the usable response-concentration relationship and help reveal nonlinearity.

Determination of Sodium by Flame Photometry

Practical 06 | Instrumental Methods of Analysis and Novel Drug Delivery Systems

Aim

To determine Sodium by Flame Photometry, and interpret the recorded observations.

Principle

- Sodium emission is compared with standards at the element-specific instrument setting. Matrix, aspiration and flame conditions influence the analytical response.
- An optical response is related to analyte concentration over the validated working range.
- A blank, suitable standards and consistent optical conditions are necessary for interpreting the response.
- Record the selected element channel and the instrument calibration.
- Use a suitable blank and consistent matrix/aspiration conditions; a different ion response is not a substitute calibration.

Materials and Apparatus

- Assigned sodium standards and test solution, flame photometer and volumetric apparatus.
- Assigned analyte, suitable reference standard, solvent/reagents and volumetric glassware.
- Specified optical instrument and compatible cells or sample-introduction system.

Method

1. Select the optical method and settings appropriate to the assigned analyte.
2. Prepare a method blank and a series of independently documented calibration concentrations.
3. Check the instrument response and measure the blank and standards using consistent settings.
4. Prepare the test solution and apply documented dilution where necessary.
5. Read the test response within the established calibration range.
6. Calculate concentration, correct for dilution and report the original-material basis.

Observation Record

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

Observation / parameter	Record / finding
Standard concentration	_____
Emission response	_____
Instrument setting	_____
Test response	_____
Dilution	_____

Calculations and Interpretation

- For calibration response $y = mC + b$: $C = (y - b) / m$.
- Original concentration = concentration read from calibration $\times$ total dilution factor.

Worked Illustration

Illustration: $y = 0.050C + 0.010$ and $y = 0.260$ give $C = 5.00$ in the concentration units used for calibration.

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

- Use clean compatible cells and consistent orientation where cells are used.
- Keep readings within the calibration range and examine the calibration fit.
- Avoid assuming that another analyte or solvent has the same response factor.

Viva Questions

1. What is the role of a blank?

It corrects the contribution of the solvent, reagents and measurement background.

2. Why use several calibration levels?

They establish the usable response-concentration relationship and help reveal nonlinearity.

Determination of Potassium by Flame Photometry

Practical 07 | Instrumental Methods of Analysis and Novel Drug Delivery Systems

Aim

To determine Potassium by Flame Photometry, and interpret the recorded observations.

Principle

- Potassium emission is compared with standards at the element-specific instrument setting. Matrix, aspiration and flame conditions influence the analytical response.
- An optical response is related to analyte concentration over the validated working range.
- A blank, suitable standards and consistent optical conditions are necessary for interpreting the response.
- Record the selected element channel and the instrument calibration.
- Use a suitable blank and consistent matrix/aspiration conditions; a different ion response is not a substitute calibration.

Materials and Apparatus

- Assigned potassium standards and test solution, flame photometer and volumetric apparatus.
- Assigned analyte, suitable reference standard, solvent/reagents and volumetric glassware.
- Specified optical instrument and compatible cells or sample-introduction system.

Method

1. Select the optical method and settings appropriate to the assigned analyte.
2. Prepare a method blank and a series of independently documented calibration concentrations.
3. Check the instrument response and measure the blank and standards using consistent settings.
4. Prepare the test solution and apply documented dilution where necessary.
5. Read the test response within the established calibration range.
6. Calculate concentration, correct for dilution and report the original-material basis.

Observation Record

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

Observation / parameter	Record / finding
Standard concentration	_____
Emission response	_____
Instrument setting	_____
Test response	_____
Dilution	_____

Calculations and Interpretation

- For calibration response $y = mC + b$: $C = (y - b) / m$.
- Original concentration = concentration read from calibration $\times$ total dilution factor.

Worked Illustration

Illustration: $y = 0.050C + 0.010$ and $y = 0.260$ give $C = 5.00$ in the concentration units used for calibration.

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

- Use clean compatible cells and consistent orientation where cells are used.
- Keep readings within the calibration range and examine the calibration fit.
- Avoid assuming that another analyte or solvent has the same response factor.

Viva Questions

1. What is the role of a blank?

It corrects the contribution of the solvent, reagents and measurement background.

2. Why use several calibration levels?

They establish the usable response-concentration relationship and help reveal nonlinearity.

Chloride and Sulphate by Nephelo-Turbidimetry

Practical 08 | Instrumental Methods of Analysis and Novel Drug Delivery Systems

Aim

To study Chloride and Sulphate by Nephelo-Turbidimetry, and interpret the recorded observations.

Principle

- The assigned chloride and sulphate reactions form dispersed particles whose optical response is compared with standards. Nephelometry measures scattered light, while turbidimetry measures loss of transmitted light.
- An optical response is related to analyte concentration over the validated working range.
- A blank, suitable standards and consistent optical conditions are necessary for interpreting the response.
- Use the precipitation system and calibration specified separately for chloride and sulphate.
- Keep mixing, timing and optical geometry comparable, since particle size and settling can alter the reading.

Materials and Apparatus

- Assigned chloride/sulphate standards and precipitation systems, suitable optical instrument and volumetric equipment.
- Assigned analyte, suitable reference standard, solvent/reagents and volumetric glassware.
- Specified optical instrument and compatible cells or sample-introduction system.

Method

1. Select the optical method and settings appropriate to the assigned analyte.
2. Prepare a method blank and a series of independently documented calibration concentrations.
3. Check the instrument response and measure the blank and standards using consistent settings.
4. Prepare the test solution and apply documented dilution where necessary.
5. Read the test response within the established calibration range.
6. Calculate concentration, correct for dilution and report the original-material basis.

Observation Record

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

Observation / parameter	Record / finding
Analyte	_____
Standard concentration	_____
Reaction/timing conditions	_____
Optical response	_____
Calculated content	_____

Calculations and Interpretation

- For calibration response $y = mC + b$: $C = (y - b) / m$.
- Original concentration = concentration read from calibration $\times$ total dilution factor.

Worked Illustration

Illustration: $y = 0.050C + 0.010$ and $y = 0.260$ give $C = 5.00$ in the concentration units used for calibration.

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

- Use clean compatible cells and consistent orientation where cells are used.
- Keep readings within the calibration range and examine the calibration fit.
- Avoid assuming that another analyte or solvent has the same response factor.

Viva Questions

1. How do nephelometry and turbidimetry differ?

They observe scattered light and attenuation of transmitted light respectively.

2. What is the role of a blank?

It corrects the contribution of the solvent, reagents and measurement background.

3. Why use several calibration levels?

They establish the usable response-concentration relationship and help reveal nonlinearity.

Separation of Sugars by Thin-Layer Chromatography

Practical 09 | Instrumental Methods of Analysis and Novel Drug Delivery Systems

Aim

To study Separation of Sugars by Thin-Layer Chromatography, and interpret the recorded observations.

Principle

- The assigned TLC system separates sugar standards and a mixture for migration-pattern comparison. Detection and R_f depend on the stationary/mobile-phase and visualization system.
- Compounds separate because they interact differently with the stationary and mobile phases.
- Identity assessment uses an appropriate comparison standard and method conditions; one migration or retention value alone is not conclusive.

Materials and Apparatus

- Prepared sugar standards/mixture, assigned TLC plate and development/detection system.
- Assigned test extract or mixture, comparison standards and the specified stationary/mobile phase system.
- Appropriate chromatographic apparatus, application equipment, developing chamber or instrument and the assigned detector.

Method

1. Record the stationary phase, mobile phase and the purpose of the separation.
2. Prepare test and comparison solutions at suitable concentrations for the assigned method.
3. Introduce measured test and comparison portions without overloading the separation system.
4. Develop or run the separation under the assigned conditions, recording the relevant operating details.
5. Locate separated bands, spots or peaks using the specified observation method.
6. Compare the test pattern with the standard and calculate the appropriate separation metric.

Observation Record

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

Observation / parameter	Record / finding
Sugar/mixture	_____
Spot appearance	_____
Spot distance	_____
Front distance	_____
Rf and match	_____

Calculations and Interpretation

- For planar chromatography: $R_f = \text{distance travelled by spot centre} / \text{distance travelled by solvent front}$.
- R_f is dimensionless and normally lies between zero and one under a valid development.

Worked Illustration

Illustration: a spot travelling 3.0 cm with a solvent front travelling 6.0 cm has $R_f = 0.50$.

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 application concentrated and avoid overloading.
- Record the solvent front immediately for planar chromatography.
- Use retention or migration data only with the stated system and relevant identity evidence.

Viva Questions

1. Why does overloading impair separation?

It broadens bands or spots and can change apparent migration or retention.

2. Why run a comparison standard?

It supplies a relevant reference pattern under the same analytical conditions.

Separation of Plant Pigments by Column Chromatography

Practical 10 | Instrumental Methods of Analysis and Novel Drug Delivery Systems

Aim

To study Separation of Plant Pigments by Column Chromatography, and interpret the recorded observations.

Principle

- Pigment bands separate because of differences in interaction with the packed stationary phase and mobile phase. A visible band colour is useful for tracking but is not complete identity evidence.
- Compounds separate because they interact differently with the stationary and mobile phases.
- Identity assessment uses an appropriate comparison standard and method conditions; one migration or retention value alone is not conclusive.
- Observe the packed bed and continuous solvent path so channel formation can be distinguished from normal band movement.
- Keep fraction labels linked to collection sequence; compare fractions using the assigned independent identity test.

Materials and Apparatus

- Prepared pigment mixture, packed teaching column, assigned mobile phase and collection vessels.
- Assigned test extract or mixture, comparison standards and the specified stationary/mobile phase system.
- Appropriate chromatographic apparatus, application equipment, developing chamber or instrument and the assigned detector.

Method

1. Record the stationary phase, mobile phase and the purpose of the separation.
2. Prepare test and comparison solutions at suitable concentrations for the assigned method.
3. Introduce measured test and comparison portions without overloading the separation system.
4. Develop or run the separation under the assigned conditions, recording the relevant operating details.
5. Locate separated bands, spots or peaks using the specified observation method.
6. Compare the test pattern with the standard and calculate the appropriate separation metric.

Observation Record

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

Observation / parameter	Record / finding
Fraction label	_____
Band colour	_____
Elution order	_____
Collected amount	_____
Comparison evidence	_____

Calculations and Interpretation

- Record fraction order, volume and the assigned identity observations.
- For any recovery calculation, use a defined starting amount and verified recovered amount.

Result

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

Finding: _____

Conclusion: _____

Precautions

- Keep application concentrated and avoid overloading.
- Record the solvent front immediately for planar chromatography.
- Use retention or migration data only with the stated system and relevant identity evidence.

Viva Questions

1. Why does overloading impair separation?

It broadens bands or spots and can change apparent migration or retention.

2. Why run a comparison standard?

It supplies a relevant reference pattern under the same analytical conditions.

High-Performance Liquid Chromatography Demonstration

Practical 11 | Instrumental Methods of Analysis and Novel Drug Delivery Systems

Aim

To study High-Performance Liquid Chromatography Demonstration, and interpret the recorded observations.

Principle

- HPLC pumps a liquid mobile phase through a column and measures separated components with the selected detector. Retention, peak area and system suitability have different roles.
- The construction and operating principle determine the function of the equipment.
- The practical distinguishes the input, working zone, controlled variables and output.
- Major parts: solvent reservoirs, degassing arrangement, pump, injector, column, detector and data system.
- Trace the flow path and identify where separation and detection occur.
- A reference calibration can relate peak response to amount; retention time alone is not sufficient identity evidence.

Materials and Apparatus

- Instructor-operated HPLC system, reference/test chromatograms and method record.
- Assigned equipment demonstration, labelled schematic or manufacturer-approved training material.
- Observation sheet and any instructor-supplied performance readings.

Method

1. Identify the major components on the actual equipment or training diagram.
2. Trace the material, energy or signal path through the system.
3. Observe the instructor demonstration and note the purpose of each control.
4. Record the operating variables and the output relevant to the exercise.
5. Relate the equipment principle to its pharmaceutical application.
6. Summarize the variables that influence performance and the checks required before operation.

Observation Record

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

Observation / parameter	Record / finding
Mobile phase	_____
Column	_____
Flow and detector setting	_____
Retention time	_____
Peak area	_____
Suitability observations	_____

Calculations and Interpretation

- For a validated calibration $y = mC + b$, concentration $C = (\text{peak response} - b)/m$.
- Apply the stated preparation/dilution correction and reference-standard potency.

Result

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

Finding: _____

Conclusion: _____

Precautions

- Use equipment demonstrations and trained operation for moving, hot or pressurized components.
- Do not bypass guards, interlocks or the assigned operating limits.
- Distinguish operating performance from the appearance of the machine.

Viva Questions

1. What does the HPLC column do?

It provides the stationary phase through which different interactions create separation.

2. Why trace the material or energy path?

It connects the equipment construction to the process it performs.

3. Why are operating variables recorded?

They are needed to explain or reproduce the observed performance.

Gas Chromatography Demonstration

Practical 12 | Instrumental Methods of Analysis and Novel Drug Delivery Systems

Aim

To study Gas Chromatography Demonstration, and interpret the recorded observations.

Principle

- GC separates sufficiently volatile, thermally suitable components carried through a column by a gas. Inlet conditions, column temperature and detector choice affect the chromatogram.
- The construction and operating principle determine the function of the equipment.
- The practical distinguishes the input, working zone, controlled variables and output.
- Major parts: carrier-gas control, injection inlet, column in a temperature-controlled oven, detector and data system.
- Explain why nonvolatile or thermally unstable compounds may be unsuitable without a separately validated preparation approach.
- Retention and detector response must be interpreted in the exact method context.

Materials and Apparatus

- Instructor-operated GC system, assigned reference/test chromatograms and method record.
- Assigned equipment demonstration, labelled schematic or manufacturer-approved training material.
- Observation sheet and any instructor-supplied performance readings.

Method

1. Identify the major components on the actual equipment or training diagram.
2. Trace the material, energy or signal path through the system.
3. Observe the instructor demonstration and note the purpose of each control.
4. Record the operating variables and the output relevant to the exercise.
5. Relate the equipment principle to its pharmaceutical application.
6. Summarize the variables that influence performance and the checks required before operation.

Observation Record

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

Observation / parameter	Record / finding
Carrier gas	_____
Inlet/column	_____
Temperature programme	_____
Detector	_____
Retention and peak response	_____

Calculations and Interpretation

- Calculate concentration using the assigned external-standard or internal-standard calibration.
- For an internal standard, retain the defined analyte/standard response and amount ratios.

Result

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

Finding: _____

Conclusion: _____

Precautions

- Use equipment demonstrations and trained operation for moving, hot or pressurized components.
- Do not bypass guards, interlocks or the assigned operating limits.
- Distinguish operating performance from the appearance of the machine.

Viva Questions

1. Why does GC use temperature control?

It affects vaporization and component partitioning, and therefore retention and separation.

2. Why trace the material or energy path?

It connects the equipment construction to the process it performs.

3. Why are operating variables recorded?

They are needed to explain or reproduce the observed performance.

Dissolution Profile of a Marketed Controlled-Release Formulation

Practical 13 | Instrumental Methods of Analysis and Novel Drug Delivery Systems

Aim

To study Dissolution Profile of a Marketed Controlled-Release Formulation, and interpret the recorded observations.

Principle

- A controlled- or sustained-release dissolution profile measures drug entering the specified medium with time. It is method-dependent and does not by itself establish in-vivo equivalence.
- 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 marketed CR/SR product, dissolution apparatus and validated 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. Record product identity, declared dose and the assigned dissolution method.
2. Check the medium, apparatus, agitation and temperature specified for that product.
3. Obtain timed aliquots in the supervised demonstration and document replacement volumes.
4. Determine concentrations using the validated analytical calibration.
5. Correct cumulative amount for drug removed in earlier aliquots when the sampling design requires it.
6. Plot percentage released against time and compare with the product-specific criterion supplied.

Observation Record

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

Observation / parameter	Record / finding
Time	_____
Medium volume	_____
Aliquot volume	_____
Assay concentration	_____
Cumulative amount	_____
Percent released	_____

Calculations and Interpretation

- With constant vessel volume V and equal withdrawn/replaced aliquot volume v : cumulative amount at time $n = V C_n + v \times \text{sum}(C_i \text{ for earlier samples})$.
- Released (%) = corrected cumulative amount/declared dose $\times 100$.
- Concentrations must represent the vessel medium after any analytical dilution correction.

Worked Illustration

Illustration: $V = 900 \text{ mL}$, $v = 5 \text{ mL}$, $C_1 = 0.020 \text{ mg/mL}$ and $C_2 = 0.040 \text{ mg/mL}$ give cumulative amount at time 2 = $900 \times 0.040 + 5 \times 0.020 = 36.1 \text{ mg}$, for equal replacement and the stated constant-volume design.

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 correct for withdrawn drug?

Earlier aliquots remove drug that would otherwise be omitted from the cumulative released amount.

2. Why control the test conditions?

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

Preparation and Evaluation of Sustained-Release Matrix Tablets

Practical 14 | Instrumental Methods of Analysis and Novel Drug Delivery Systems

Aim

To study Preparation and Evaluation of Sustained-Release Matrix Tablets, and interpret the recorded observations.

Principle

- A matrix can control release through diffusion, swelling, erosion or combined processes. Tablet mechanical properties, drug content and release profile are evaluated separately.
- A dosage form combines an active ingredient with excipients selected for processing, stability and performance.
- Preparation and evaluation are linked: changes in composition or process can alter the product quality attributes.
- Identify the matrix polymer and the proposed release mechanism.
- Observe blend/granulation and compression in the approved laboratory process.
- Assay extracted drug by UV against a suitable calibration.
- Measure the assigned dissolution profile and relate it to the matrix design.

Materials and Apparatus

- Assigned drug, matrix polymer and excipients; laboratory tablet preparation and UV-assay equipment.
- Assigned active ingredient, excipients and the instructor-approved master formula.
- Balance, mixing/preparation equipment, packaging materials and the assigned evaluation apparatus.

Method

1. Read the master formula and identify the purpose of each ingredient.
2. Calculate the quantities for the assigned laboratory batch and check ingredient identity.
3. Follow the specified order of addition and preparation process, recording actual quantities and process observations.
4. Measure the yield or finished quantity and package the laboratory preparation as specified.
5. Evaluate the product attributes listed for this dosage form with documented methods.
6. Compare the observations with the assigned specification and explain any difference.

Observation Record

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

Observation / parameter	Record / finding
Polymer/formula	_____
Tablet dimensions	_____
Mechanical observations	_____
Drug content	_____
Time-release profile	_____

Calculations and Interpretation

- Assay (%) = $\text{measured active amount} / \text{declared active amount} \times 100$.
- For release measurements, apply the actual sampling and dilution corrections before calculating percentage released.

Result

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

Finding: _____

Conclusion: _____

Precautions

- Record changes in process or composition; do not conceal a deviation.
- Use the specification assigned to the exact product and dosage form.
- A teaching batch is for laboratory evaluation and is not released for patient use.

Viva Questions

1. Why identify excipient functions?

Each excipient can influence processing, stability, release or acceptability.

2. Why is appearance alone insufficient for evaluation?

It does not establish content, performance, stability or microbiological quality.

Preparation and Evaluation of Nanoparticles with HPLC Drug Assay

Practical 15 | Instrumental Methods of Analysis and Novel Drug Delivery Systems

Aim

To study Preparation and Evaluation of Nanoparticles with HPLC Drug Assay, and interpret the recorded observations.

Principle

- Nanoparticle formulation involves a carrier system whose size and drug association require separate measurements. HPLC can quantify extracted drug, but it does not directly determine particle diameter.
- A dosage form combines an active ingredient with excipients selected for processing, stability and performance.
- Preparation and evaluation are linked: changes in composition or process can alter the product quality attributes.
- Identify the carrier and the broad particle-formation method demonstrated.
- Record independent size/distribution observations such as the assigned light-scattering result.
- Review the separation and extraction recovery used for drug-association measurements.
- Quantify drug by HPLC and retain the exact denominator for loading or encapsulation.

Materials and Apparatus

- Instructor-approved carrier formulation, prepared dispersions, separation/size records and HPLC calibration.
- Assigned active ingredient, excipients and the instructor-approved master formula.
- Balance, mixing/preparation equipment, packaging materials and the assigned evaluation apparatus.

Method

1. Read the master formula and identify the purpose of each ingredient.
2. Calculate the quantities for the assigned laboratory batch and check ingredient identity.
3. Follow the specified order of addition and preparation process, recording actual quantities and process observations.
4. Measure the yield or finished quantity and package the laboratory preparation as specified.
5. Evaluate the product attributes listed for this dosage form with documented methods.
6. Compare the observations with the assigned specification and explain any difference.

Observation Record

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

Observation / parameter	Record / finding
Carrier/formula	_____
Size and distribution method	_____
Extracted-drug response	_____
Free/associated drug	_____
Loading measure	_____

Calculations and Interpretation

- Encapsulation efficiency (%) = $\frac{\text{associated drug amount}}{\text{total drug amount introduced}} \times 100$.
- Drug loading (%) = $\frac{\text{associated drug mass}}{\text{defined recovered formulation mass}} \times 100$.
- State whether the loading denominator is carrier mass or total dried product mass.

Result

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

Finding: _____

Conclusion: _____

Precautions

- Record changes in process or composition; do not conceal a deviation.
- Use the specification assigned to the exact product and dosage form.
- A teaching batch is for laboratory evaluation and is not released for patient use.

Viva Questions

1. Why identify excipient functions?

Each excipient can influence processing, stability, release or acceptability.

2. Why is appearance alone insufficient for evaluation?

It does not establish content, performance, stability or microbiological quality.

Preparation and Evaluation of Liposomes

Practical 16 | Instrumental Methods of Analysis and Novel Drug Delivery Systems

Aim

To study Preparation and Evaluation of Liposomes, and interpret the recorded observations.

Principle

- Liposomes are phospholipid-bilayer vesicles. Drug association, vesicle size, dispersion properties and leakage/stability require distinct measurements.
- A dosage form combines an active ingredient with excipients selected for processing, stability and performance.
- Preparation and evaluation are linked: changes in composition or process can alter the product quality attributes.
- Identify the lipid and aqueous components in the approved formulation.
- Observe the vesicle-preparation demonstration and record the composition/process variables.
- Review the method used to distinguish unentrapped from vesicle-associated drug.
- Evaluate size and drug association separately and discuss the limitations of the measurements.

Materials and Apparatus

- Prepared lipid-vesicle teaching formulations, separation records and assigned drug/size assays.
- Assigned active ingredient, excipients and the instructor-approved master formula.
- Balance, mixing/preparation equipment, packaging materials and the assigned evaluation apparatus.

Method

1. Read the master formula and identify the purpose of each ingredient.
2. Calculate the quantities for the assigned laboratory batch and check ingredient identity.
3. Follow the specified order of addition and preparation process, recording actual quantities and process observations.
4. Measure the yield or finished quantity and package the laboratory preparation as specified.
5. Evaluate the product attributes listed for this dosage form with documented methods.
6. Compare the observations with the assigned specification and explain any difference.

Observation Record

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

Observation / parameter	Record / finding
Lipid composition	_____
Vesicle-size method	_____
Total drug	_____
Unentrapped drug	_____
Entrapment	_____
Stability observations	_____

Calculations and Interpretation

- Entrapped amount = measured total drug - measured unentrapped drug, after valid separation/recovery corrections.
- Entrapment efficiency (%) = $\frac{\text{entrapped amount}}{\text{total drug amount}} \times 100$.

Worked Illustration

Illustration: total drug 10.0 mg and verified unentrapped drug 2.0 mg give entrapped drug 8.0 mg and entrapment efficiency 80.0%.

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

- Record changes in process or composition; do not conceal a deviation.
- Use the specification assigned to the exact product and dosage form.
- A teaching batch is for laboratory evaluation and is not released for patient use.

Viva Questions

1. Why identify excipient functions?

Each excipient can influence processing, stability, release or acceptability.

2. Why is appearance alone insufficient for evaluation?

It does not establish content, performance, stability or microbiological quality.

Preparation and Evaluation of Buccal Dosage Form with UV Evaluation

Practical 17 | Instrumental Methods of Analysis and Novel Drug Delivery Systems

Aim

To study Preparation and Evaluation of Buccal Dosage Form with UV Evaluation, and interpret the recorded observations.

Principle

- A buccal film or tablet uses a suitable polymer system for mucosal contact. Drug content, mechanical properties, swelling/adhesion and release are separate attributes.
- A dosage form combines an active ingredient with excipients selected for processing, stability and performance.
- Preparation and evaluation are linked: changes in composition or process can alter the product quality attributes.
- Identify polymer, plasticizer and active-ingredient functions.
- Observe the assigned preparation and record finished unit dimensions/mass.
- Extract a defined unit for UV assay with suitable blank/interference checks.
- Evaluate the assigned swelling/adhesion and in-vitro release measures.

Materials and Apparatus

- Approved drug/polymer formula, laboratory preparation equipment and UV-assay/release apparatus.
- Assigned active ingredient, excipients and the instructor-approved master formula.
- Balance, mixing/preparation equipment, packaging materials and the assigned evaluation apparatus.

Method

1. Read the master formula and identify the purpose of each ingredient.
2. Calculate the quantities for the assigned laboratory batch and check ingredient identity.
3. Follow the specified order of addition and preparation process, recording actual quantities and process observations.
4. Measure the yield or finished quantity and package the laboratory preparation as specified.
5. Evaluate the product attributes listed for this dosage form with documented methods.
6. Compare the observations with the assigned specification and explain any difference.

Observation Record

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

Observation / parameter	Record / finding
Formula and unit size	_____
Thickness or strength	_____
Swelling/adhesion	_____
Extracted content	_____
Release profile	_____

Calculations and Interpretation

- Assay (%) = measured active amount/declared active amount × 100.
- For release measurements, apply the actual sampling and dilution corrections before calculating percentage released.

Result

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

Finding: _____

Conclusion: _____

Precautions

- Record changes in process or composition; do not conceal a deviation.
- Use the specification assigned to the exact product and dosage form.
- A teaching batch is for laboratory evaluation and is not released for patient use.

Viva Questions

1. Why identify excipient functions?

Each excipient can influence processing, stability, release or acceptability.

2. Why is appearance alone insufficient for evaluation?

It does not establish content, performance, stability or microbiological quality.

Preparation and Evaluation of Paracetamol Transdermal Patch with UV Evaluation

Practical 18 | Instrumental Methods of Analysis and Novel Drug Delivery Systems

Aim

To study Preparation and Evaluation of Paracetamol Transdermal Patch with UV Evaluation, and interpret the recorded observations.

Principle

- A polymeric patch study examines drug loading, film properties and in-vitro release. Release into a medium is different from skin permeation, and a laboratory film is not automatically a clinically effective transdermal system.
- A dosage form combines an active ingredient with excipients selected for processing, stability and performance.
- Preparation and evaluation are linked: changes in composition or process can alter the product quality attributes.
- Identify the active, polymer, plasticizer and backing arrangement in the assigned design.
- Observe preparation and define the patch area used for testing.
- Assay extracted paracetamol with blank/interference and recovery considerations.
- Evaluate film properties and in-vitro release, reporting permeation only if a separate valid permeation study is supplied.

Materials and Apparatus

- Approved paracetamol/polymer laboratory formula, film-preparation equipment and UV-assay/release setup.
- Assigned active ingredient, excipients and the instructor-approved master formula.
- Balance, mixing/preparation equipment, packaging materials and the assigned evaluation apparatus.

Method

1. Read the master formula and identify the purpose of each ingredient.
2. Calculate the quantities for the assigned laboratory batch and check ingredient identity.
3. Follow the specified order of addition and preparation process, recording actual quantities and process observations.
4. Measure the yield or finished quantity and package the laboratory preparation as specified.
5. Evaluate the product attributes listed for this dosage form with documented methods.
6. Compare the observations with the assigned specification and explain any difference.

Observation Record

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

Observation / parameter	Record / finding
Film area	_____
Thickness/mass	_____
Extracted paracetamol	_____
Area loading	_____
Release profile	_____
Film integrity	_____

Calculations and Interpretation

- Area loading = measured drug amount/tested film area.
- Released (%) = corrected cumulative drug amount/initial drug amount in the tested patch × 100.

Worked Illustration

Illustration: a tested film area of 4.00 cm² containing 8.00 mg paracetamol has area loading 2.00 mg/cm².

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

- Record changes in process or composition; do not conceal a deviation.
- Use the specification assigned to the exact product and dosage form.
- A teaching batch is for laboratory evaluation and is not released for patient use.

Viva Questions

1. Why identify excipient functions?

Each excipient can influence processing, stability, release or acceptability.

2. Why is appearance alone insufficient for evaluation?

It does not establish content, performance, stability or microbiological quality.

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