Pharm.D - Year 3

Pharmaceutical Analysis

Complete subject manual: 22 practicals and activities. Each chapter includes a student observation or work record.

Use the PDF bookmarks to navigate. The table gives each practical's content-page count; the combined manual has continuous page numbers.

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Amino Acids by Paper Chromatography

Experiment 1 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To separate and compare the assigned compounds by amino acids by paper chromatography.

Principle

Differential distribution in the paper/mobile-phase system produces different migration of the assigned amino acids.

Requirements

- Assigned chromatography paper and development chamber.
- Assigned standards, coded mixture, prescribed mobile phase, application tools and visualisation method.

Procedure

1. Plan the separation

Record the analytes, stationary phase, mobile-phase composition and prescribed visualisation method.

2. Apply the material

Mark an origin in pencil and apply small compact standard and mixture spots; allow each application to dry as assigned.

3. Develop

Place the paper in the prepared prescribed solvent system with the origin above the liquid, and allow the assigned development.

4. Visualise and measure

Mark the front, dry and apply the prescribed amino-acid visualisation method under supervision; record spot positions and colours.

5. Compare

Compare mixture spots with standards on the same run. Record number of spots, migration and limitations; matching migration alone is not absolute proof of identity.

Observations and Record

Lane / spot	Distance from origin	Rf / appearance
Standard 1	_____	_____
Standard 2	_____	_____
Assigned mixture	_____	_____
Solvent front	_____	Not applicable

Worked Example

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

- Rf = distance travelled by the spot centre / distance travelled by the solvent front, both measured from the same origin.
- Example: 4.0 cm / 8.0 cm = 0.50. Compare only under the same separation conditions.

Result

Record the completed observations, calculations and conclusion with units. State any limitation of the observation.

Precautions

- Keep the origin above the solvent level and mark the front promptly.
- Use small compact applications and the authorised ventilation/visualisation precautions.

Viva Questions and Answers

1. Why compare standards on the same run?

Migration depends on the separation conditions.

2. What can a second spot indicate?

A mixture, impurity or other separated component; interpretation needs the method context.

Sulphonamides by Thin-layer Chromatography

Experiment 2 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To separate and compare the assigned compounds by sulphonamides by thin-layer chromatography.

Principle

Adsorption and distribution between the stationary layer and mobile phase separate the assigned sulphonamides.

Requirements

- Assigned TLC plate and chamber.
- Assigned standards, coded mixture, prescribed mobile phase, application tools and visualisation method.

Procedure

1. Plan the separation

Record the analytes, stationary phase, mobile-phase composition and prescribed visualisation method.

2. Apply the material

Use a pencil origin and apply compact standard and test spots without damaging the layer.

3. Develop

Develop in the assigned mobile phase with the application points above the solvent; protect the plate from uneven wetting.

4. Visualise and measure

Mark the front, dry and use the assigned UV or chemical detection method; record each spot and migration.

5. Compare

Compare mixture spots with standards on the same run. Record number of spots, migration and limitations; matching migration alone is not absolute proof of identity.

Observations and Record

Lane / spot	Distance from origin	Rf / appearance
Standard 1	_____	_____
Standard 2	_____	_____
Assigned mixture	_____	_____
Solvent front	_____	Not applicable

Worked Example

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

- Rf = distance travelled by the spot centre / distance travelled by the solvent front, both measured from the same origin.
- Example: 4.0 cm / 8.0 cm = 0.50. Compare only under the same separation conditions.

Result

Record the completed observations, calculations and conclusion with units. State any limitation of the observation.

Precautions

- Keep the origin above the solvent level and mark the front promptly.
- Use small compact applications and the authorised ventilation/visualisation precautions.

Viva Questions and Answers

1. Why compare standards on the same run?

Migration depends on the separation conditions.

2. What can a second spot indicate?

A mixture, impurity or other separated component; interpretation needs the method context.

pH and Solvent Effects on UV Spectra

Experiment 3 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To compare the UV spectra of an assigned compound under specified pH and solvent conditions.

Principle

Ionisation and solvation can alter electronic absorption. A valid comparison uses matched concentration, appropriate blanks and a defined wavelength range.

Requirements

- Assigned UV-visible instrument, suitable matched cells, analyte, prescribed solvent/buffer systems and blanks.

Procedure

1. Set the comparison

Record each solvent/pH condition and keep analyte concentration and optical path constant.

2. Blank separately

Use the corresponding solvent/buffer blank and a suitable spectral range for that medium.

3. Scan

Record spectra and locate maxima and selected absorbances without exceeding the validated range.

4. Compare

Tabulate wavelength/absorbance changes, distinguishing wavelength shift from intensity change.

5. Interpret

Relate the pattern to the assigned ionisation or solvent explanation and state any precipitation or baseline limitation.

Observations and Record

Condition	Wavelength maximum	Selected absorbance
Condition 1	_____	_____
Condition 2	_____	_____
Condition 3	_____	_____

Result

Record the completed observations, calculations and conclusion with units. State any limitation of the observation.

Precautions

- Do not attribute concentration or baseline differences to a solvent effect.
- Record actual pH and any turbidity or cell mismatch.

Viva Questions and Answers

1. What is a bathochromic shift?

A shift of absorption to longer wavelength.

2. Why use a separate blank?

Different solvents and buffers contribute different backgrounds.

Derivative UV Spectroscopy

Experiment 4 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To determine the assigned drug by validated derivative-UV calibration.

Principle

A derivative spectrum describes the rate of absorbance change with wavelength. It can improve separation of overlapping spectral features when a validated derivative method is used.

Requirements

- Assigned test material and the prescribed method reagents or standards.
- Calibrated volumetric glassware, appropriate instrument and record sheet.

Procedure

1. Prepare and identify the test

Confirm the analyte, method version, concentration basis and test-portion amount. Prepare the test solution as specified and record any dilution.

2. Set the measurement basis

Check the instrument or standardised titrant. Prepare the specified blank, standard and control so that the test and comparison use the same basis.

3. Make the measurement

Apply the prescribed validated derivative-UV calibration method and record assigned derivative amplitude at the stated wavelength. Record replicate readings and any interfering appearance.

4. Calculate

Apply the stated equation with the correct blank correction, dilution and unit conversion. Show the substitution and retain the observed readings.

5. Assess the result

Compare replicate agreement and the assigned specification. Record a result with units and the limitations of the method.

Observations and Record

Measurement	Reading
Test portion / dilution	_____
Blank / standard	_____
assigned derivative amplitude at the stated wavelength	_____
Calculated content and unit	_____

Worked Example

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

- For an assigned linear calibration D = mC + b, C = (D − b)/m. Apply all dilution factors.
- Example: D = 0.030, b = 0.002 and m = 0.004 per microgram/mL give 7.0 micrograms/mL. Record derivative order, wavelength interval and processing parameters.

Result

Record the completed observations, calculations and conclusion with units. State any limitation of the observation.

Precautions

- Use the exact method reagents and assigned endpoint; different methods are not interchangeable.
- Record the blank correction and the actual test portion rather than an assumed value.
- Hazardous reagent operations require the specified laboratory facilities and supervision.

Viva Questions and Answers

1. What produces the analytical signal?

A derivative spectrum describes the rate of absorbance change with wavelength. It can improve separation of overlapping spectral features when a validated derivative method is used.

2. Why include a blank or control?

It identifies contributions unrelated to the analyte or checks that the measurement system performed appropriately.

3. What must accompany the result?

Units, method, dilution basis, relevant control performance and any limitation.

Indicator pKa by UV Spectroscopy

Experiment 5 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To estimate the pKa of an assigned indicator using absorbance at known pH.

Principle

The acid and base indicator forms have different spectra. At a suitable wavelength, absorbance can estimate their ratio if total concentration and path length are constant.

Requirements

- Assigned indicator, buffers, pH meter, UV-visible instrument, matched cells and appropriate blanks.

Procedure

1. Obtain end-form readings

Record the assigned acid-form and base-form absorbances at the same wavelength and total concentration.

2. Measure intermediate pH

Read actual pH and absorbance for the assigned buffer conditions.

3. Calculate the ratio

Use the two-form absorbance relation only if Beer-Lambert behaviour and the endpoint forms are suitable.

4. Calculate pKa

Apply the acid/base equation and compare estimates from more than one intermediate point.

5. Record limits

Avoid an estimate from absorbance indistinguishable from either end-form value.

Observations and Record

Reading	Value
Acid-form absorbance A_HA	_____
Base-form absorbance A_A	_____
Intermediate pH / absorbance A	_____
Calculated ratio / pKa	_____

Worked Example

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

- [A−]/[HA] = (A − A_HA)/(A_A − A), for matched total concentration and path.
- pKa = pH − log10([A−]/[HA]).
- Example: A_HA = 0.10, A_A = 0.90, A = 0.50 and pH = 7.0 give a ratio of 1 and pKa = 7.0.

Result

Record the completed observations, calculations and conclusion with units. State any limitation of the observation.

Precautions

- Use measured pH and valid endpoint-form spectra.
- Do not use the relation if precipitation or additional absorbing species invalidate the two-form model.

Viva Questions and Answers

1. What happens when acid and base forms are equal?

pH equals pKa in the ideal stated acid/base model.

2. Why avoid the extreme end-form points?

Small absorbance errors can produce large ratio errors near an endpoint.

Conductometric Titration of a Mixed Acid

Experiment 6 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To interpret the conductometric titration of the assigned strong/weak acid mixture.

Principle

Conductivity changes as ions are consumed and replaced. Changes in slope can locate equivalence regions when the assigned mixture gives distinguishable stages.

Requirements

- Conductivity meter/cell, assigned acid mixture, standardised base and calibrated titration equipment.

Procedure

1. Prepare the measurement

Check the cell and record temperature, test aliquot and titrant concentration.

2. Record titration data

Follow the prescribed additions and record conductivity and cumulative volume after stabilisation.

3. Apply the stated correction

Use the method-prescribed dilution correction where needed and plot corrected response against volume.

4. Locate stages

Fit the assigned straight-line regions and obtain their intersections; do not choose an endpoint solely from a single noisy reading.

5. Resolve the mixture

Use the validated first and total equivalence definitions for the specified strong/weak acid mixture.

Observations and Record

Cumulative volume	Conductivity	Correction / region
_____	_____	_____
_____	_____	_____
_____	_____	_____

Worked Example

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

- For the assigned monoprotic strong + weak acid model: strong-acid moles = Cbase × V1(L); weak-acid moles = Cbase × [V2 − V1](L).
- Example: 0.1 mol/L base, V1 = 10 mL and V2 = 25 mL give 0.001 mol strong acid and 0.0015 mol weak acid.

Result

Record the completed observations, calculations and conclusion with units. State any limitation of the observation.

Precautions

- Keep temperature and cell placement consistent.
- Use the specified mixture model; endpoints may not be resolvable for every acid combination.

Viva Questions and Answers

1. Why does slope change?

The ionic species and their contribution to conductivity change during neutralisation.

2. Why record temperature?

Conductivity is temperature dependent.

Potentiometric Acid-base Titration

Experiment 7 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To locate an acid-base equivalence point from measured pH or potential.

Principle

An electrode measures a potential related to the solution state. A titration curve and its slope locate an equivalence region without relying on a visual colour endpoint.

Requirements

- Calibrated pH/potential system, assigned analyte, standardised titrant and volumetric equipment.

Procedure

1. Calibrate and record

Use the prescribed electrode calibration, temperature and test aliquot.

2. Titrate

Add the assigned titrant portions, mix and record a stable pH/potential at each cumulative volume.

3. Plot

Plot reading against volume and examine the first-derivative or prescribed endpoint plot.

4. Find equivalence

Identify the endpoint from the stated analysis and compare replicates.

5. Calculate

Apply reaction stoichiometry and report concentration on the test-portion basis.

Observations and Record

Volume	pH / potential	Change per volume
_____	_____	_____
_____	_____	_____
_____	_____	_____

Worked Example

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

- For a 1:1 reaction: Ctest = Ctitrant × Veq / Vtest, using equal volume units.
- Example: 0.1 mol/L × 15.0 mL / 20.0 mL = 0.075 mol/L.

Result

Record the completed observations, calculations and conclusion with units. State any limitation of the observation.

Precautions

- Allow electrode stabilisation and record cumulative volume accurately.
- Do not confuse the largest measured pH with the equivalence point.

Viva Questions and Answers

1. How does a derivative help?

It highlights a rapid change near the equivalence region.

2. Why use stoichiometry?

The concentration relation depends on the reacting mole ratio.

Drug Assay by Fluorimetry

Experiment 8 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To determine the assigned fluorescent drug by fluorescence calibration.

Principle

A suitable analyte fluoresces under specified excitation and emission conditions. A calibrated response is useful only in its validated concentration range.

Requirements

- Assigned test material and the prescribed method reagents or standards.
- Calibrated volumetric glassware, appropriate instrument and record sheet.

Procedure

1. Prepare and identify the test

Confirm the analyte, method version, concentration basis and test-portion amount. Prepare the test solution as specified and record any dilution.

2. Set the measurement basis

Check the instrument or standardised titrant. Prepare the specified blank, standard and control so that the test and comparison use the same basis.

3. Make the measurement

Apply the prescribed fluorescence calibration method and record blank-corrected fluorescence signal. Record replicate readings and any interfering appearance.

4. Calculate

Apply the stated equation with the correct blank correction, dilution and unit conversion. Show the substitution and retain the observed readings.

5. Assess the result

Compare replicate agreement and the assigned specification. Record a result with units and the limitations of the method.

Observations and Record

Measurement	Reading
Test portion / dilution	_____
Blank / standard	_____
blank-corrected fluorescence signal	_____
Calculated content and unit	_____

Worked Example

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

- For F = mC + b, C = (F − b)/m; include test dilution.
- Example: F = 55, b = 5 and m = 10 signal units per microgram/mL give 5 micrograms/mL before dilution correction. Record excitation/emission wavelengths and matrix.

Result

Record the completed observations, calculations and conclusion with units. State any limitation of the observation.

Precautions

- Use the exact method reagents and assigned endpoint; different methods are not interchangeable.
- Record the blank correction and the actual test portion rather than an assumed value.
- Hazardous reagent operations require the specified laboratory facilities and supervision.

Viva Questions and Answers

1. What produces the analytical signal?

A suitable analyte fluoresces under specified excitation and emission conditions. A calibrated response is useful only in its validated concentration range.

2. Why include a blank or control?

It identifies contributions unrelated to the analyte or checks that the measurement system performed appropriately.

3. What must accompany the result?

Units, method, dilution basis, relevant control performance and any limitation.

Fluorescence Quenching

Experiment 9 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To compare an assigned fluorophore response with and without a quencher.

Principle

Quenching reduces observed fluorescence through specified physical or chemical mechanisms. Absorption or matrix changes can also alter the measured signal.

Requirements

- Assigned fluorophore/quencher system, fluorimeter, blanks and supplied or measured comparison data.

Procedure

1. Prepare the comparison

Keep fluorophore concentration and optical conditions matched across the assigned quencher levels.

2. Read signals

Record blank-corrected unquenched F0 and quenched F values.

3. Plot

Plot the assigned quenching relation; inspect whether it is linear over the chosen range.

4. Assess mechanism limits

Discuss inner-filter or other instrumental effects and avoid assigning a mechanism from one ratio alone.

Observations and Record

Quencher concentration	F	F0/F
0	_____	_____
_____	_____	_____
_____	_____	_____

Worked Example

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

- For the applicable Stern-Volmer model: F0/F = 1 + Ksv[Q].
- Example: F0 = 100, F = 80 and [Q] = 0.01 mol/L give Ksv = (1.25 − 1)/0.01 = 25 L/mol, if that model is valid.

Result

Record the completed observations, calculations and conclusion with units. State any limitation of the observation.

Precautions

- Use matching blanks and inspect absorption-related interference.
- Model fit alone does not establish one quenching mechanism.

Viva Questions and Answers

1. What does F0 mean?

The fluorescence without quencher under the matched conditions.

2. Why can a quencher appear effective for the wrong reason?

It may alter absorption or optical transmission rather than only the fluorophore process.

Sulphonamide Colour Assay - Bratton-Marshall Reaction

Experiment 10 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To determine the assigned sulphonamide by Bratton-Marshall colour calibration.

Principle

The assigned aromatic-amine sulphonamide is diazotised and coupled to form a coloured product under the prescribed analytical method.

Requirements

- Assigned test material and the prescribed method reagents or standards.
- Calibrated volumetric glassware, appropriate instrument and record sheet.

Procedure

1. Prepare and identify the test

Confirm the analyte, method version, concentration basis and test-portion amount. Prepare the test solution as specified and record any dilution.

2. Set the measurement basis

Check the instrument or standardised titrant. Prepare the specified blank, standard and control so that the test and comparison use the same basis.

3. Make the measurement

Apply the prescribed Bratton-Marshall colour calibration method and record absorbance of the assigned coupled product. Record replicate readings and any interfering appearance.

4. Calculate

Apply the stated equation with the correct blank correction, dilution and unit conversion. Show the substitution and retain the observed readings.

5. Assess the result

Compare replicate agreement and the assigned specification. Record a result with units and the limitations of the method.

Observations and Record

Measurement	Reading
Test portion / dilution	_____
Blank / standard	_____
absorbance of the assigned coupled product	_____
Calculated content and unit	_____

Worked Example

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

- Ctest = (Atest − Ablank)/(Astandard − Ablank) × Cstandard × dilution factor, only for a validated proportional response.
- Example: corrected responses 0.30 and 0.50 with a 10 micrograms/mL standard give 6 micrograms/mL before dilution correction.

Result

Record the completed observations, calculations and conclusion with units. State any limitation of the observation.

Precautions

- Use the exact method reagents and assigned endpoint; different methods are not interchangeable.
- Record the blank correction and the actual test portion rather than an assumed value.
- Hazardous reagent operations require the specified laboratory facilities and supervision.

Viva Questions and Answers

1. What produces the analytical signal?

The assigned aromatic-amine sulphonamide is diazotised and coupled to form a coloured product under the prescribed analytical method.

2. Why include a blank or control?

It identifies contributions unrelated to the analyte or checks that the measurement system performed appropriately.

3. What must accompany the result?

Units, method, dilution basis, relevant control performance and any limitation.

Simultaneous UV Estimation of Two Drugs

Experiment 11 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To resolve two absorbing components using a validated two-wavelength method.

Principle

At two suitable wavelengths, additive absorbances give two independent equations when each component obeys the calibrated response and interference is controlled.

Requirements

- Assigned two-drug mixture, pure standards, UV-visible instrument, matched cells and blank.

Procedure

1. Calibrate components

Determine each component response coefficient at each assigned wavelength on the same concentration/path basis.

2. Measure the mixture

Read mixture absorbance at both wavelengths after appropriate blank correction.

3. Solve

Use the simultaneous equations and check that the coefficient system is sufficiently distinct.

4. Verify

Reconstruct the predicted absorbances and compare with the measurements; report dilution-corrected component concentrations.

Observations and Record

Coefficient / reading	Value
a11 and a12 at wavelength 1	_____
a21 and a22 at wavelength 2	_____
Mixture A1 and A2	_____
Calculated C1 and C2	_____

Worked Example

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

- A1 = a11 C1 + a12 C2; A2 = a21 C1 + a22 C2.
- C1 = (A1 a22 − A2 a12)/(a11 a22 − a21 a12); C2 = (a11 A2 − a21 A1)/(a11 a22 − a21 a12).
- Example: A1 = 0.25, A2 = 0.25; coefficients a11 = 0.02, a12 = 0.01, a21 = 0.01, a22 = 0.03 give C1 = 10 and C2 = 5 in the assigned concentration units.

Result

Record the completed observations, calculations and conclusion with units. State any limitation of the observation.

Precautions

- Use calibrated coefficients with consistent units and path length.
- A nearly zero determinant makes the estimate unstable; other absorbing ingredients invalidate a simple two-component model.

Viva Questions and Answers

1. Why use two wavelengths?

They provide two independent measurements for two unknown concentrations.

2. How can the answer be checked?

Insert calculated concentrations into both equations and compare predicted with observed absorbance.

Salicylic Acid by Colorimetry

Experiment 12 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To determine salicylic acid by the assigned colorimetric method.

Principle

The prescribed salicylate colour reaction produces an analytical signal compared with standards under matched conditions.

Requirements

- Assigned test material and the prescribed method reagents or standards.
- Calibrated volumetric glassware, appropriate instrument and record sheet.

Procedure

1. Prepare and identify the test

Confirm the analyte, method version, concentration basis and test-portion amount. Prepare the test solution as specified and record any dilution.

2. Set the measurement basis

Check the instrument or standardised titrant. Prepare the specified blank, standard and control so that the test and comparison use the same basis.

3. Make the measurement

Apply the prescribed the assigned colorimetric method method and record blank-corrected absorbance at the method wavelength. Record replicate readings and any interfering appearance.

4. Calculate

Apply the stated equation with the correct blank correction, dilution and unit conversion. Show the substitution and retain the observed readings.

5. Assess the result

Compare replicate agreement and the assigned specification. Record a result with units and the limitations of the method.

Observations and Record

Measurement	Reading
Test portion / dilution	_____
Blank / standard	_____
blank-corrected absorbance at the method wavelength	_____
Calculated content and unit	_____

Worked Example

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

- C = (A − b)/m for the validated calibration A = mC + b; apply dilution factors.
- Example: A = 0.40, b = 0.05, m = 0.07 per microgram/mL give 5 micrograms/mL. Record reagent and timing conditions from the assigned method.

Result

Record the completed observations, calculations and conclusion with units. State any limitation of the observation.

Precautions

- Use the exact method reagents and assigned endpoint; different methods are not interchangeable.
- Record the blank correction and the actual test portion rather than an assumed value.
- Hazardous reagent operations require the specified laboratory facilities and supervision.

Viva Questions and Answers

1. What produces the analytical signal?

The prescribed salicylate colour reaction produces an analytical signal compared with standards under matched conditions.

2. Why include a blank or control?

It identifies contributions unrelated to the analyte or checks that the measurement system performed appropriately.

3. What must accompany the result?

Units, method, dilution basis, relevant control performance and any limitation.

Chloride and Sulphate in Calcium Gluconate by Nepheloturbidimetry

Experiment 13 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To determine chloride and sulphate-related test responses by the separately prescribed nephelometric or turbidimetric methods.

Principle

Controlled formation of an insoluble dispersed phase permits an optical comparison. Nephelometry measures scattered light; turbidimetry assesses transmitted-light attenuation.

Requirements

- Assigned test material and the prescribed method reagents or standards.
- Calibrated volumetric glassware, appropriate instrument and record sheet.

Procedure

1. Prepare and identify the test

Confirm the analyte, method version, concentration basis and test-portion amount. Prepare the test solution as specified and record any dilution.

2. Set the measurement basis

Check the instrument or standardised titrant. Prepare the specified blank, standard and control so that the test and comparison use the same basis.

3. Make the measurement

Apply the prescribed the separately prescribed nephelometric or turbidimetric methods method and record blank and standard-comparison response for each ion. Record replicate readings and any interfering appearance.

4. Calculate

Apply the stated equation with the correct blank correction, dilution and unit conversion. Show the substitution and retain the observed readings.

5. Assess the result

Compare replicate agreement and the assigned specification. Record a result with units and the limitations of the method.

Observations and Record

Measurement	Reading
Test portion / dilution	_____
Blank / standard	_____
blank and standard-comparison response for each ion	_____
Calculated content and unit	_____

Worked Example

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

- Use the method-specific calibration and dilution basis for each ion. For a linear teaching calibration $S = mC + b$, $C = (S - b)/m$.
- Example: $S = 0.35$, $b = 0.05$ and $m = 0.10$ give $C = 3$ assigned units. Chloride and sulphate require their own standards and reagents.

Result

Record the completed observations, calculations and conclusion with units. State any limitation of the observation.

Precautions

- Use the exact method reagents and assigned endpoint; different methods are not interchangeable.
- Record the blank correction and the actual test portion rather than an assumed value.
- Hazardous reagent operations require the specified laboratory facilities and supervision.

Viva Questions and Answers

1. What produces the analytical signal?

Controlled formation of an insoluble dispersed phase permits an optical comparison. Nephelometry measures scattered light; turbidimetry assesses transmitted-light attenuation.

2. Why include a blank or control?

It identifies contributions unrelated to the analyte or checks that the measurement system performed appropriately.

3. What must accompany the result?

Units, method, dilution basis, relevant control performance and any limitation.

Sodium and Potassium by Flame Photometry

Experiment 14 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To determine sodium and potassium separately by flame-photometric calibration.

Principle

Atomised sodium and potassium emit element-specific light under the defined measurement conditions. Standards must match the analytical basis and relevant matrix.

Requirements

- Assigned test material and the prescribed method reagents or standards.
- Calibrated volumetric glassware, appropriate instrument and record sheet.

Procedure

1. Prepare and identify the test

Confirm the analyte, method version, concentration basis and test-portion amount. Prepare the test solution as specified and record any dilution.

2. Set the measurement basis

Check the instrument or standardised titrant. Prepare the specified blank, standard and control so that the test and comparison use the same basis.

3. Make the measurement

Apply the prescribed flame-photometric calibration method and record element-channel signal and standards. Record replicate readings and any interfering appearance.

4. Calculate

Apply the stated equation with the correct blank correction, dilution and unit conversion. Show the substitution and retain the observed readings.

5. Assess the result

Compare replicate agreement and the assigned specification. Record a result with units and the limitations of the method.

Observations and Record

Measurement	Reading
Test portion / dilution	_____
Blank / standard	_____
element-channel signal and standards	_____
Calculated content and unit	_____

Worked Example

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

- $C = (S - b)/m \times$ dilution factor for each separately calibrated channel.
- Example: $S = 45$, $b = 5$ and $m = 2$ give 20 assigned concentration units before dilution correction. Do not share one calibration between Na and K.

Result

Record the completed observations, calculations and conclusion with units. State any limitation of the observation.

Precautions

- Use the exact method reagents and assigned endpoint; different methods are not interchangeable.
- Record the blank correction and the actual test portion rather than an assumed value.
- Hazardous reagent operations require the specified laboratory facilities and supervision.

Viva Questions and Answers

1. What produces the analytical signal?

Atomised sodium and potassium emit element-specific light under the defined measurement conditions. Standards must match the analytical basis and relevant matrix.

2. Why include a blank or control?

It identifies contributions unrelated to the analyte or checks that the measurement system performed appropriately.

3. What must accompany the result?

Units, method, dilution basis, relevant control performance and any limitation.

pKa by pH-metric Titration

Experiment 15 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To estimate the pKa of an assigned weak acid from its titration curve.

Principle

For the applicable monoprotic weak-acid model, pH at half-neutralisation approximates pKa when the stated assumptions are suitable.

Requirements

- Assigned weak acid, standardised base, calibrated pH meter and titration equipment.

Procedure

1. Record conditions

Enter acid identity, test aliquot, titrant and temperature.

2. Collect data

Record stabilised pH at the assigned cumulative titrant volumes.

3. Find equivalence

Use the prescribed titration-curve analysis to locate the relevant equivalence volume.

4. Find half-equivalence

Read/interpolate pH at half that volume and assess the model assumptions.

Observations and Record

Reading	Value
Equivalence volume	_____
Half-equivalence volume	_____
pH at half-equivalence	_____
pKa estimate and limitation	_____

Worked Example

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

- For the stated weak-acid model, pKa ≈ pH at Veq/2.
- Example: Veq = 20 mL, so read pH at 10 mL; a pH of 4.8 gives an approximate pKa of 4.8.

Result

Record the completed observations, calculations and conclusion with units. State any limitation of the observation.

Precautions

- Do not use one half-equivalence rule for overlapping polyprotic stages without the assigned model.
- Use measured pH, not a nominal buffer value.

Viva Questions and Answers

1. Why is half-equivalence useful?

The acid and conjugate-base amounts are equal in the ideal model.

2. What limits the estimate?

Activity effects, concentration, temperature and unsuitable or unresolved titration stages.

Specific Rotation by Polarimetry

Experiment 16 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To determine specific rotation of the assigned material by polarimetry.

Principle

An optically active material rotates plane-polarised light; the result depends on wavelength, temperature, path length, concentration and solvent.

Requirements

- Assigned test material and the prescribed method reagents or standards.
- Calibrated volumetric glassware, appropriate instrument and record sheet.

Procedure

1. Prepare and identify the test

Confirm the analyte, method version, concentration basis and test-portion amount. Prepare the test solution as specified and record any dilution.

2. Set the measurement basis

Check the instrument or standardised titrant. Prepare the specified blank, standard and control so that the test and comparison use the same basis.

3. Make the measurement

Apply the prescribed polarimetry method and record blank-corrected observed rotation. Record replicate readings and any interfering appearance.

4. Calculate

Apply the stated equation with the correct blank correction, dilution and unit conversion. Show the substitution and retain the observed readings.

5. Assess the result

Compare replicate agreement and the assigned specification. Record a result with units and the limitations of the method.

Observations and Record

Measurement	Reading
Test portion / dilution	_____
Blank / standard	_____
blank-corrected observed rotation	_____
Calculated content and unit	_____

Worked Example

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

- [alpha] = alpha/(l × c), using l in dm and c in g/mL; if c is expressed as g/100 mL, use [alpha] = 100 alpha/(l × c).
- Example: alpha = +2.0 degrees, l = 1 dm and c = 0.020 g/mL give a specific rotation of +100 on that stated basis.

Result

Record the completed observations, calculations and conclusion with units. State any limitation of the observation.

Precautions

- Use the exact method reagents and assigned endpoint; different methods are not interchangeable.
- Record the blank correction and the actual test portion rather than an assumed value.
- Hazardous reagent operations require the specified laboratory facilities and supervision.

Viva Questions and Answers

1. What produces the analytical signal?

An optically active material rotates plane-polarised light; the result depends on wavelength, temperature, path length, concentration and solvent.

2. Why include a blank or control?

It identifies contributions unrelated to the analyte or checks that the measurement system performed appropriately.

3. What must accompany the result?

Units, method, dilution basis, relevant control performance and any limitation.

IR Spectra of a Compound and Its Derivative

Experiment 17 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To compare the supplied parent and derivative spectra.

Principle

Infrared absorption provides functional-group and fingerprint evidence. A spectrum must be compared with the specified compound and measurement mode.

Requirements

- Instructor-provided labelled diagrams, traces or data for this activity.
- Calculator, graph paper or spreadsheet, and the activity record.

Procedure

1. Read axes

Record wavenumber units, absorbance/transmittance display and measurement mode.

2. Identify groups

Mark assigned diagnostic carbonyl, hydroxyl, amino or other relevant bands without treating approximate ranges as exact identity tests.

3. Compare derivative

Explain the expected group change and whether the supplied spectrum supports it.

4. Assess identity

Compare the full pattern with the assigned reference, considering preparation and instrument conditions.

Observations and Record

Record item	Entry
Activity / material identifier	_____
Measurement or visible feature	_____
Comparison / calculated outcome	_____
Interpretation and limitation	_____

Result

The recorded finding is ____; the supporting observation or calculation is ____. The main limitation is ____.

Precautions

- Use the supplied teaching material; distinguish a measured observation from an assumed value.
- Keep units, axes, labels and measurement conditions consistent.
- Record limitations and seek instructor review before applying a conclusion to a live case.

Viva Questions and Answers

1. Why use the fingerprint region?

Its combined pattern can support comparison beyond a single functional-group band.

2. Does one carbonyl band name a compound?

No. Many compounds share that functional group.

HPLC - Instrument Demonstration

Experiment 18 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To interpret the supplied hplc - instrument demonstration.

Principle

A liquid mobile phase carries components through a stationary phase; retention and calibrated detector response support separation and quantification.

Requirements

- Instructor-provided labelled diagrams, traces or data for this activity.
- Calculator, graph paper or spreadsheet, and the activity record.

Procedure

1. Label flow path

Identify solvent reservoirs, pump, injector, column, detector and data system.

2. Review the method

Record column, mobile phase, flow, detector and standards from the supplied run.

3. Read the chromatogram

Record retention times, peak areas, resolution and system-suitability findings.

4. Interpret

Compare standard and test evidence; retention alone does not conclusively establish identity.

Observations and Record

Record item	Entry
Activity / material identifier	_____
Measurement or visible feature	_____
Comparison / calculated outcome	_____
Interpretation and limitation	_____

Worked Example

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

- For a suitable area calibration Area = mC + b, C = (Area − b)/m.
- Example: area 1100, b = 100 and m = 200 give C = 5 assigned units before dilution.

Result

The recorded finding is ____; the supporting observation or calculation is ____. The main limitation is ____.

Precautions

- Use the supplied teaching material; distinguish a measured observation from an assumed value.
- Keep units, axes, labels and measurement conditions consistent.
- Record limitations and seek instructor review before applying a conclusion to a live case.

Viva Questions and Answers

1. What is the main endpoint?

Interpret the supplied hplc - instrument demonstration.

2. Does one observed result prove a general conclusion?

No. Controls, repeatability and the limitations of the method must be considered.

HPTLC - Instrument Demonstration

Experiment 19 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To interpret the supplied hptlc - instrument demonstration.

Principle

Planar chromatographic separation with controlled application and scanning produces migration and response records.

Requirements

- Instructor-provided labelled diagrams, traces or data for this activity.
- Calculator, graph paper or spreadsheet, and the activity record.

Procedure

1. Identify equipment

Label application device, plate, chamber and scanner in the demonstration.

2. Read conditions

Record plate, mobile phase, development and detection settings.

3. Read tracks

Compare standard/test migration, peak pattern and scan response.

4. Assess

State identity or quantity evidence only when the assigned method and controls support it.

Observations and Record

Record item	Entry
Activity / material identifier	_____
Measurement or visible feature	_____
Comparison / calculated outcome	_____
Interpretation and limitation	_____

Worked Example

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

- Rf = spot migration / solvent-front migration.
- Example: 30 mm / 60 mm = 0.50.

Result

The recorded finding is ____; the supporting observation or calculation is ____. The main limitation is ____.

Precautions

- Use the supplied teaching material; distinguish a measured observation from an assumed value.
- Keep units, axes, labels and measurement conditions consistent.
- Record limitations and seek instructor review before applying a conclusion to a live case.

Viva Questions and Answers

1. What is the main endpoint?

Interpret the supplied hptlc - instrument demonstration.

2. Does one observed result prove a general conclusion?

No. Controls, repeatability and the limitations of the method must be considered.

GC-MS - Instrument Demonstration

Experiment 20 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To interpret the supplied gc-ms - instrument demonstration.

Principle

Gas-chromatographic separation and mass-spectral detection provide complementary retention and ion-pattern evidence for suitable volatile analytes.

Requirements

- Instructor-provided labelled diagrams, traces or data for this activity.
- Calculator, graph paper or spreadsheet, and the activity record.

Procedure

1. Label the system

Identify carrier-gas system, injector, column, oven, interface and mass analyser.

2. Read supplied settings

Record sample preparation, chromatographic method and ionisation mode from the demonstration.

3. Compare evidence

Use retention and the supplied mass spectrum to assess the assigned candidate.

4. Record limits

A library match is supporting evidence; coelution, background and instrument conditions require review.

Observations and Record

Record item	Entry
Activity / material identifier	_____
Measurement or visible feature	_____
Comparison / calculated outcome	_____
Interpretation and limitation	_____

Result

The recorded finding is ____; the supporting observation or calculation is ____. The main limitation is ____.

Precautions

- Use the supplied teaching material; distinguish a measured observation from an assumed value.
- Keep units, axes, labels and measurement conditions consistent.
- Record limitations and seek instructor review before applying a conclusion to a live case.

Viva Questions and Answers

1. What is the main endpoint?

Interpret the supplied gc-ms - instrument demonstration.

2. Does one observed result prove a general conclusion?

No. Controls, repeatability and the limitations of the method must be considered.

DSC - Thermal-analysis Demonstration

Experiment 21 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To interpret the supplied dsc - thermal-analysis demonstration.

Principle

Differential scanning calorimetry records a heat-flow difference during a defined thermal programme. Transitions depend on the material and programme.

Requirements

- Instructor-provided labelled diagrams, traces or data for this activity.
- Calculator, graph paper or spreadsheet, and the activity record.

Procedure

1. Read conditions

Record specimen mass, pan, atmosphere and heating/cooling programme from the supplied trace.

2. Locate events

Mark assigned endothermic/exothermic direction, onset, peak and any baseline step.

3. Classify cautiously

Distinguish a melting event from a glass-transition-like baseline change using the assigned comparison.

4. Report

Record transition temperatures and assigned enthalpy with the calculation basis; do not infer purity from a single peak.

Observations and Record

Record item	Entry
Activity / material identifier	_____
Measurement or visible feature	_____
Comparison / calculated outcome	_____
Interpretation and limitation	_____

Worked Example

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

- Mass-normalised enthalpy = integrated heat / specimen mass.
- Example: 0.10 J / 0.002 g = 50 J/g on the supplied integration basis.

Result

The recorded finding is ____; the supporting observation or calculation is ____. The main limitation is ____.

Precautions

- Use the supplied teaching material; distinguish a measured observation from an assumed value.
- Keep units, axes, labels and measurement conditions consistent.
- Record limitations and seek instructor review before applying a conclusion to a live case.

Viva Questions and Answers

1. What is the main endpoint?

Interpret the supplied dsc - thermal-analysis demonstration.

2. Does one observed result prove a general conclusion?

No. Controls, repeatability and the limitations of the method must be considered.

NMR Spectrum Interpretation

Experiment 22 - Practical Notes

Name: _____ Roll no.: _____ Date: _____

Aim

To interpret the supplied nmr spectrum interpretation.

Principle

Chemical shift, integral and splitting describe nuclei in a molecular environment. Reliable interpretation uses the nucleus, solvent and the assigned spectrum conditions.

Requirements

- Instructor-provided labelled diagrams, traces or data for this activity.
- Calculator, graph paper or spreadsheet, and the activity record.

Procedure

1. Read the spectrum

Record nucleus, chemical-shift scale, solvent and supplied frequency.

2. Assign signals

Mark chemically distinct signals and compare integral ratios for appropriate non-exchangeable signals.

3. Analyse splitting

Use the assigned simple coupling model where suitable; complex spectra are not resolved by a universal n+1 rule.

4. Compare structure

Check whether the proposed structure accounts for the full pattern, integration and known exchange/overlap limitations.

Observations and Record

Record item	Entry
Activity / material identifier	_____
Measurement or visible feature	_____
Comparison / calculated outcome	_____
Interpretation and limitation	_____

Worked Example

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

- Integral ratios support relative proton numbers in the applicable 1H spectrum.
- Example: clean integral values 2:3 support a 2:3 relative proton ratio, subject to the stated integration conditions.

Result

The recorded finding is ____; the supporting observation or calculation is ____. The main limitation is ____.

Precautions

- Use the supplied teaching material; distinguish a measured observation from an assumed value.
- Keep units, axes, labels and measurement conditions consistent.
- Record limitations and seek instructor review before applying a conclusion to a live case.

Viva Questions and Answers

1. What is the main endpoint?

Interpret the supplied nmr spectrum interpretation.

2. Does one observed result prove a general conclusion?

No. Controls, repeatability and the limitations of the method must be considered.

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