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# Semester 4 - Practical Notes

## Medicinal Chemistry I

### Complete Subject Manual

16 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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# Preparation of Pyrazole Ring

Practical 01 | Medicinal Chemistry I

## Aim

To prepare Pyrazole Ring, and interpret the recorded observations.

## Principle

- Pyrazoles are five-membered aromatic rings with adjacent nitrogen atoms. Identify the exact assigned derivative before drawing a reaction; the ring is not described correctly by placing its nitrogens at positions 1 and 3.
- A chemical transformation changes selected functional groups while retaining or constructing the required molecular skeleton.
- Product identity and purity require analytical evidence; appearance alone cannot establish either.
- Reaction record: draw starting material and product structures, mark the functional-group change, and retain the assigned stoichiometry.
- Characterization record: compare the assigned melting range, TLC or spectral evidence. A narrow melting interval can support purity but cannot alone establish complete identity.

## Materials and Apparatus

- Instructor-selected precursor system and demonstrated pyrazole derivative.
- Assigned starting materials and instructor-selected reagents.
- Balance, reaction glassware, stirring/heating arrangement, condenser where required, filtration equipment and identity/purity test apparatus.

## Method

1. Write the assigned reaction scheme and balance the transformation before calculating reagent requirements.
2. Identify the limiting reactant and document the approved reaction scale, solvent and conditions.
3. Observe the instructor-directed reaction sequence and record changes without substituting reagents or conditions.
4. Complete the prescribed work-up to separate the product from reaction liquids and by-products.
5. Purify the product by the assigned method, dry it appropriately and determine the recovered mass.
6. Record the assigned identity and purity observations and compare them with the expected product.

## Observation Record

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

| Observation / parameter      | Record / finding |
|------------------------------|------------------|
| Assigned derivative          | _____            |
| Reaction scheme              | _____            |
| Limiting-reactant record     | _____            |
| Dry recovery                 | _____            |
| Identity/purity observations | _____            |

## Calculations and Interpretation

- Theoretical product mass = limiting-reactant amount (mol)  $\times$  product stoichiometric ratio  $\times$  product molar mass.
- Percentage yield = dry product mass / theoretical product mass  $\times$  100.

### Worked Illustration

Illustration: a theoretical product mass of 2.00 g and dry recovery of 1.50 g give 75.0% yield.

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 the approved reaction conditions and suitable ventilation.
- Keep product, starting material and waste streams separately labelled.
- Calculate yield from dry product and the limiting reactant; never use wet mass as pure-product mass.

## Viva Questions

### 1. What is the limiting reactant?

The reactant whose available amount permits the smallest theoretical amount of product.

### 2. Does a high yield establish purity?

No. Residual solvent, impurities and unreacted material can increase recovered mass.

# Preparation of Oxazole Ring

Practical 02 | Medicinal Chemistry I

## Aim

To prepare Oxazole Ring, and interpret the recorded observations.

## Principle

- Oxazole is a five-membered aromatic ring containing oxygen at position 1 and nitrogen at position 3. Ring-forming chemistry must be tied to the exact assigned derivative.
- A chemical transformation changes selected functional groups while retaining or constructing the required molecular skeleton.
- Product identity and purity require analytical evidence; appearance alone cannot establish either.
- Reaction record: draw starting material and product structures, mark the functional-group change, and retain the assigned stoichiometry.
- Characterization record: compare the assigned melting range, TLC or spectral evidence. A narrow melting interval can support purity but cannot alone establish complete identity.

## Materials and Apparatus

- Instructor-selected oxygen/nitrogen precursor system.
- Assigned starting materials and instructor-selected reagents.
- Balance, reaction glassware, stirring/heating arrangement, condenser where required, filtration equipment and identity/purity test apparatus.

## Method

1. Write the assigned reaction scheme and balance the transformation before calculating reagent requirements.
2. Identify the limiting reactant and document the approved reaction scale, solvent and conditions.
3. Observe the instructor-directed reaction sequence and record changes without substituting reagents or conditions.
4. Complete the prescribed work-up to separate the product from reaction liquids and by-products.
5. Purify the product by the assigned method, dry it appropriately and determine the recovered mass.
6. Record the assigned identity and purity observations and compare them with the expected product.

## Observation Record

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

| Observation / parameter      | Record / finding |
|------------------------------|------------------|
| Assigned derivative          | _____            |
| Reaction scheme              | _____            |
| Limiting-reactant record     | _____            |
| Dry recovery                 | _____            |
| Identity/purity observations | _____            |

## Calculations and Interpretation

- Theoretical product mass = limiting-reactant amount (mol)  $\times$  product stoichiometric ratio  $\times$  product molar mass.
- Percentage yield = dry product mass / theoretical product mass  $\times$  100.

### Worked Illustration

Illustration: a theoretical product mass of 2.00 g and dry recovery of 1.50 g give 75.0% yield.

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 the approved reaction conditions and suitable ventilation.
- Keep product, starting material and waste streams separately labelled.
- Calculate yield from dry product and the limiting reactant; never use wet mass as pure-product mass.

## Viva Questions

### 1. What is the limiting reactant?

The reactant whose available amount permits the smallest theoretical amount of product.

### 2. Does a high yield establish purity?

No. Residual solvent, impurities and unreacted material can increase recovered mass.

# Preparation of Benzimidazole

Practical 03 | Medicinal Chemistry I

## Aim

To prepare Benzimidazole, and interpret the recorded observations.

## Principle

- Benzimidazole formation constructs an imidazole ring fused to benzene. The assigned condensation/cyclization connects an ortho-diamino aromatic system to a suitable carbon source.
- A chemical transformation changes selected functional groups while retaining or constructing the required molecular skeleton.
- Product identity and purity require analytical evidence; appearance alone cannot establish either.
- Reaction record: draw starting material and product structures, mark the functional-group change, and retain the assigned stoichiometry.
- Characterization record: compare the assigned melting range, TLC or spectral evidence. A narrow melting interval can support purity but cannot alone establish complete identity.

## Materials and Apparatus

- Assigned ortho-diamino starting material and carbon-source reagent.
- Assigned starting materials and instructor-selected reagents.
- Balance, reaction glassware, stirring/heating arrangement, condenser where required, filtration equipment and identity/purity test apparatus.

## Method

1. Write the assigned reaction scheme and balance the transformation before calculating reagent requirements.
2. Identify the limiting reactant and document the approved reaction scale, solvent and conditions.
3. Observe the instructor-directed reaction sequence and record changes without substituting reagents or conditions.
4. Complete the prescribed work-up to separate the product from reaction liquids and by-products.
5. Purify the product by the assigned method, dry it appropriately and determine the recovered mass.
6. Record the assigned identity and purity observations and compare them with the expected product.

## Observation Record

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

| Observation / parameter      | Record / finding |
|------------------------------|------------------|
| Assigned derivative          | _____            |
| Reaction scheme              | _____            |
| Limiting-reactant record     | _____            |
| Dry recovery                 | _____            |
| Identity/purity observations | _____            |

## Calculations and Interpretation

- Theoretical product mass = limiting-reactant amount (mol)  $\times$  product stoichiometric ratio  $\times$  product molar mass.
- Percentage yield = dry product mass / theoretical product mass  $\times$  100.

### Worked Illustration

Illustration: a theoretical product mass of 2.00 g and dry recovery of 1.50 g give 75.0% yield.

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 the approved reaction conditions and suitable ventilation.
- Keep product, starting material and waste streams separately labelled.
- Calculate yield from dry product and the limiting reactant; never use wet mass as pure-product mass.

## Viva Questions

### 1. What is the limiting reactant?

The reactant whose available amount permits the smallest theoretical amount of product.

### 2. Does a high yield establish purity?

No. Residual solvent, impurities and unreacted material can increase recovered mass.

# Preparation of Benzotriazole

Practical 04 | Medicinal Chemistry I

## Aim

To prepare Benzotriazole, and interpret the recorded observations.

## Principle

- Benzotriazole contains a fused three-nitrogen heterocycle. The assigned ring-forming demonstration must be interpreted by tracking the nitrogen atoms and the aromatic skeleton.
- A chemical transformation changes selected functional groups while retaining or constructing the required molecular skeleton.
- Product identity and purity require analytical evidence; appearance alone cannot establish either.
- Reaction record: draw starting material and product structures, mark the functional-group change, and retain the assigned stoichiometry.
- Characterization record: compare the assigned melting range, TLC or spectral evidence. A narrow melting interval can support purity but cannot alone establish complete identity.

## Materials and Apparatus

- Instructor-demonstrated benzotriazole ring-forming materials.
- Assigned starting materials and instructor-selected reagents.
- Balance, reaction glassware, stirring/heating arrangement, condenser where required, filtration equipment and identity/purity test apparatus.

## Method

1. Write the assigned reaction scheme and balance the transformation before calculating reagent requirements.
2. Identify the limiting reactant and document the approved reaction scale, solvent and conditions.
3. Observe the instructor-directed reaction sequence and record changes without substituting reagents or conditions.
4. Complete the prescribed work-up to separate the product from reaction liquids and by-products.
5. Purify the product by the assigned method, dry it appropriately and determine the recovered mass.
6. Record the assigned identity and purity observations and compare them with the expected product.

## Observation Record

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

| Observation / parameter      | Record / finding |
|------------------------------|------------------|
| Assigned derivative          | _____            |
| Reaction scheme              | _____            |
| Limiting-reactant record     | _____            |
| Dry recovery                 | _____            |
| Identity/purity observations | _____            |

## Calculations and Interpretation

- Theoretical product mass = limiting-reactant amount (mol)  $\times$  product stoichiometric ratio  $\times$  product molar mass.
- Percentage yield = dry product mass / theoretical product mass  $\times$  100.

### Worked Illustration

Illustration: a theoretical product mass of 2.00 g and dry recovery of 1.50 g give 75.0% yield.

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 the approved reaction conditions and suitable ventilation.
- Keep product, starting material and waste streams separately labelled.
- Calculate yield from dry product and the limiting reactant; never use wet mass as pure-product mass.

## Viva Questions

### 1. What is the limiting reactant?

The reactant whose available amount permits the smallest theoretical amount of product.

### 2. Does a high yield establish purity?

No. Residual solvent, impurities and unreacted material can increase recovered mass.

# Preparation of 2,3-Diphenylquinoxaline

Practical 05 | Medicinal Chemistry I

## Aim

To prepare 2,3-Diphenylquinoxaline, and interpret the recorded observations.

## Principle

- Condensation of an ortho-diamine with an adjacent diketone constructs the quinoxaline ring. The phenyl substituents originate from the assigned diketone.
- A chemical transformation changes selected functional groups while retaining or constructing the required molecular skeleton.
- Product identity and purity require analytical evidence; appearance alone cannot establish either.
- Reaction record: draw starting material and product structures, mark the functional-group change, and retain the assigned stoichiometry.
- Characterization record: compare the assigned melting range, TLC or spectral evidence. A narrow melting interval can support purity but cannot alone establish complete identity.

## Materials and Apparatus

- Assigned aromatic ortho-diamine and diphenyl diketone.
- Assigned starting materials and instructor-selected reagents.
- Balance, reaction glassware, stirring/heating arrangement, condenser where required, filtration equipment and identity/purity test apparatus.

## Method

1. Write the assigned reaction scheme and balance the transformation before calculating reagent requirements.
2. Identify the limiting reactant and document the approved reaction scale, solvent and conditions.
3. Observe the instructor-directed reaction sequence and record changes without substituting reagents or conditions.
4. Complete the prescribed work-up to separate the product from reaction liquids and by-products.
5. Purify the product by the assigned method, dry it appropriately and determine the recovered mass.
6. Record the assigned identity and purity observations and compare them with the expected product.

## Observation Record

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

| Observation / parameter      | Record / finding |
|------------------------------|------------------|
| Assigned derivative          | _____            |
| Reaction scheme              | _____            |
| Limiting-reactant record     | _____            |
| Dry recovery                 | _____            |
| Identity/purity observations | _____            |

## Calculations and Interpretation

- Theoretical product mass = limiting-reactant amount (mol)  $\times$  product stoichiometric ratio  $\times$  product molar mass.
- Percentage yield = dry product mass / theoretical product mass  $\times$  100.

### Worked Illustration

Illustration: a theoretical product mass of 2.00 g and dry recovery of 1.50 g give 75.0% yield.

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 the approved reaction conditions and suitable ventilation.
- Keep product, starting material and waste streams separately labelled.
- Calculate yield from dry product and the limiting reactant; never use wet mass as pure-product mass.

## Viva Questions

### 1. What is the limiting reactant?

The reactant whose available amount permits the smallest theoretical amount of product.

### 2. Does a high yield establish purity?

No. Residual solvent, impurities and unreacted material can increase recovered mass.

# Preparation of Benzocaine

Practical 06 | Medicinal Chemistry I

## Aim

To prepare Benzocaine, and interpret the recorded observations.

## Principle

- Benzocaine is ethyl para-aminobenzoate. The assigned ester-forming route retains the para amino group and introduces the ethyl ester functionality.
- A chemical transformation changes selected functional groups while retaining or constructing the required molecular skeleton.
- Product identity and purity require analytical evidence; appearance alone cannot establish either.
- Reaction record: draw starting material and product structures, mark the functional-group change, and retain the assigned stoichiometry.
- Characterization record: compare the assigned melting range, TLC or spectral evidence. A narrow melting interval can support purity but cannot alone establish complete identity.

## Materials and Apparatus

- Assigned para-aminobenzoic-acid/ester-forming demonstration materials.
- Assigned starting materials and instructor-selected reagents.
- Balance, reaction glassware, stirring/heating arrangement, condenser where required, filtration equipment and identity/purity test apparatus.

## Method

1. Write the assigned reaction scheme and balance the transformation before calculating reagent requirements.
2. Identify the limiting reactant and document the approved reaction scale, solvent and conditions.
3. Observe the instructor-directed reaction sequence and record changes without substituting reagents or conditions.
4. Complete the prescribed work-up to separate the product from reaction liquids and by-products.
5. Purify the product by the assigned method, dry it appropriately and determine the recovered mass.
6. Record the assigned identity and purity observations and compare them with the expected product.

## Observation Record

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

| Observation / parameter      | Record / finding |
|------------------------------|------------------|
| Assigned derivative          | _____            |
| Reaction scheme              | _____            |
| Limiting-reactant record     | _____            |
| Dry recovery                 | _____            |
| Identity/purity observations | _____            |

## Calculations and Interpretation

- Theoretical product mass = limiting-reactant amount (mol)  $\times$  product stoichiometric ratio  $\times$  product molar mass.
- Percentage yield = dry product mass / theoretical product mass  $\times$  100.

### Worked Illustration

Illustration: a theoretical product mass of 2.00 g and dry recovery of 1.50 g give 75.0% yield.

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 the approved reaction conditions and suitable ventilation.
- Keep product, starting material and waste streams separately labelled.
- Calculate yield from dry product and the limiting reactant; never use wet mass as pure-product mass.

## Viva Questions

### 1. What is the limiting reactant?

The reactant whose available amount permits the smallest theoretical amount of product.

### 2. Does a high yield establish purity?

No. Residual solvent, impurities and unreacted material can increase recovered mass.

# Preparation of Phenytoin

Practical 07 | Medicinal Chemistry I

## Aim

To prepare Phenytoin, and interpret the recorded observations.

## Principle

- Phenytoin has a substituted hydantoin ring. The assigned ring-forming sequence is interpreted by tracking carbonyl and nitrogen functionality into that ring.
- A chemical transformation changes selected functional groups while retaining or constructing the required molecular skeleton.
- Product identity and purity require analytical evidence; appearance alone cannot establish either.
- Reaction record: draw starting material and product structures, mark the functional-group change, and retain the assigned stoichiometry.
- Characterization record: compare the assigned melting range, TLC or spectral evidence. A narrow melting interval can support purity but cannot alone establish complete identity.

## Materials and Apparatus

- Instructor-demonstrated hydantoin-forming precursor system.
- Assigned starting materials and instructor-selected reagents.
- Balance, reaction glassware, stirring/heating arrangement, condenser where required, filtration equipment and identity/purity test apparatus.

## Method

1. Write the assigned reaction scheme and balance the transformation before calculating reagent requirements.
2. Identify the limiting reactant and document the approved reaction scale, solvent and conditions.
3. Observe the instructor-directed reaction sequence and record changes without substituting reagents or conditions.
4. Complete the prescribed work-up to separate the product from reaction liquids and by-products.
5. Purify the product by the assigned method, dry it appropriately and determine the recovered mass.
6. Record the assigned identity and purity observations and compare them with the expected product.

## Observation Record

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

| Observation / parameter      | Record / finding |
|------------------------------|------------------|
| Assigned derivative          | _____            |
| Reaction scheme              | _____            |
| Limiting-reactant record     | _____            |
| Dry recovery                 | _____            |
| Identity/purity observations | _____            |

## Calculations and Interpretation

- Theoretical product mass = limiting-reactant amount (mol)  $\times$  product stoichiometric ratio  $\times$  product molar mass.
- Percentage yield = dry product mass / theoretical product mass  $\times$  100.

### Worked Illustration

Illustration: a theoretical product mass of 2.00 g and dry recovery of 1.50 g give 75.0% yield.

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 the approved reaction conditions and suitable ventilation.
- Keep product, starting material and waste streams separately labelled.
- Calculate yield from dry product and the limiting reactant; never use wet mass as pure-product mass.

## Viva Questions

### 1. What is the limiting reactant?

The reactant whose available amount permits the smallest theoretical amount of product.

### 2. Does a high yield establish purity?

No. Residual solvent, impurities and unreacted material can increase recovered mass.

# Preparation of Phenothiazine

Practical 08 | Medicinal Chemistry I

## Aim

To prepare Phenothiazine, and interpret the recorded observations.

## Principle

- Phenothiazine is a tricyclic nitrogen/sulfur heterocycle. Distinguish the parent ring system from medicinal derivatives bearing side chains or additional substituents.
- A chemical transformation changes selected functional groups while retaining or constructing the required molecular skeleton.
- Product identity and purity require analytical evidence; appearance alone cannot establish either.
- Reaction record: draw starting material and product structures, mark the functional-group change, and retain the assigned stoichiometry.
- Characterization record: compare the assigned melting range, TLC or spectral evidence. A narrow melting interval can support purity but cannot alone establish complete identity.

## Materials and Apparatus

- Instructor-demonstrated phenothiazine ring-system materials.
- Assigned starting materials and instructor-selected reagents.
- Balance, reaction glassware, stirring/heating arrangement, condenser where required, filtration equipment and identity/purity test apparatus.

## Method

1. Write the assigned reaction scheme and balance the transformation before calculating reagent requirements.
2. Identify the limiting reactant and document the approved reaction scale, solvent and conditions.
3. Observe the instructor-directed reaction sequence and record changes without substituting reagents or conditions.
4. Complete the prescribed work-up to separate the product from reaction liquids and by-products.
5. Purify the product by the assigned method, dry it appropriately and determine the recovered mass.
6. Record the assigned identity and purity observations and compare them with the expected product.

## Observation Record

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

| Observation / parameter      | Record / finding |
|------------------------------|------------------|
| Assigned derivative          | _____            |
| Reaction scheme              | _____            |
| Limiting-reactant record     | _____            |
| Dry recovery                 | _____            |
| Identity/purity observations | _____            |

## Calculations and Interpretation

- Theoretical product mass = limiting-reactant amount (mol)  $\times$  product stoichiometric ratio  $\times$  product molar mass.
- Percentage yield = dry product mass / theoretical product mass  $\times$  100.

### Worked Illustration

Illustration: a theoretical product mass of 2.00 g and dry recovery of 1.50 g give 75.0% yield.

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 the approved reaction conditions and suitable ventilation.
- Keep product, starting material and waste streams separately labelled.
- Calculate yield from dry product and the limiting reactant; never use wet mass as pure-product mass.

## Viva Questions

### 1. What is the limiting reactant?

The reactant whose available amount permits the smallest theoretical amount of product.

### 2. Does a high yield establish purity?

No. Residual solvent, impurities and unreacted material can increase recovered mass.

# Preparation of Barbiturate Ring System

Practical 09 | Medicinal Chemistry I

## Aim

To prepare Barbiturate Ring System, and interpret the recorded observations.

## Principle

- The barbiturate nucleus is a pyrimidine-trione system. Structural study must distinguish the parent scaffold from a specific substituted medicinal compound.
- A chemical transformation changes selected functional groups while retaining or constructing the required molecular skeleton.
- Product identity and purity require analytical evidence; appearance alone cannot establish either.
- Reaction record: draw starting material and product structures, mark the functional-group change, and retain the assigned stoichiometry.
- Characterization record: compare the assigned melting range, TLC or spectral evidence. A narrow melting interval can support purity but cannot alone establish complete identity.

## Materials and Apparatus

- Assigned structure model and instructor-demonstrated ring-forming teaching material.
- Assigned starting materials and instructor-selected reagents.
- Balance, reaction glassware, stirring/heating arrangement, condenser where required, filtration equipment and identity/purity test apparatus.

## Method

1. Write the assigned reaction scheme and balance the transformation before calculating reagent requirements.
2. Identify the limiting reactant and document the approved reaction scale, solvent and conditions.
3. Observe the instructor-directed reaction sequence and record changes without substituting reagents or conditions.
4. Complete the prescribed work-up to separate the product from reaction liquids and by-products.
5. Purify the product by the assigned method, dry it appropriately and determine the recovered mass.
6. Record the assigned identity and purity observations and compare them with the expected product.

## Observation Record

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

| Observation / parameter      | Record / finding |
|------------------------------|------------------|
| Assigned derivative          | _____            |
| Reaction scheme              | _____            |
| Limiting-reactant record     | _____            |
| Dry recovery                 | _____            |
| Identity/purity observations | _____            |

## Calculations and Interpretation

- Theoretical product mass = limiting-reactant amount (mol)  $\times$  product stoichiometric ratio  $\times$  product molar mass.
- Percentage yield = dry product mass / theoretical product mass  $\times$  100.

### Worked Illustration

Illustration: a theoretical product mass of 2.00 g and dry recovery of 1.50 g give 75.0% yield.

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 the approved reaction conditions and suitable ventilation.
- Keep product, starting material and waste streams separately labelled.
- Calculate yield from dry product and the limiting reactant; never use wet mass as pure-product mass.

## Viva Questions

### 1. What is the limiting reactant?

The reactant whose available amount permits the smallest theoretical amount of product.

### 2. Does a high yield establish purity?

No. Residual solvent, impurities and unreacted material can increase recovered mass.

# Assay of Chlorpromazine

Practical 10 | Medicinal Chemistry I

## Aim

To assay Chlorpromazine, and interpret the recorded observations.

## Principle

- Basic functionality permits a suitable assigned nonaqueous acid-base assay; the salt form and specified endpoint determine the calculation basis.
- A standardized titrant reacts with the analyte according to a defined stoichiometric relationship.
- The calculation must distinguish direct titration from a blank-corrected or back-titration procedure.

## Materials and Apparatus

- Assigned chlorpromazine form, standardized assay titrant and specified solvent/endpoint system.
- Assigned test material, standardized titrant, specified solvent and indicator or endpoint detector.
- Burette, pipette, volumetric flask, conical flask and analytical balance.

## Method

1. Identify the analyte form, the reaction and the endpoint specified for the exercise.
2. Document titrant concentration or factor and prepare a reagent blank where the method requires one.
3. Measure the prescribed test portion and dissolve or treat it by the assigned analytical method.
4. Titrate to the specified endpoint while recording initial and final burette readings.
5. Repeat the determination with independently measured portions and retain all readings.
6. Apply blank, dilution and stoichiometric corrections before reporting the result.

## Observation Record

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

| Observation / parameter | Record / finding |
|-------------------------|------------------|
| Analyte form            | _____            |
| Test mass               | _____            |
| Blank reading           | _____            |
| Test reading            | _____            |
| Method factor           | _____            |
| Assay percentage        | _____            |

## Calculations and Interpretation

- Method-corrected analyte mass = corrected titrant volume × verified analyte factor × dilution correction.
- Assay (%) = method-corrected analyte mass/test-portion mass × 100.
- Use the exact salt/hydrate and reaction factor assigned to this assay.

## Result

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

Finding: \_\_\_\_\_

Conclusion: \_\_\_\_\_

## Precautions

- Condition volumetric apparatus and avoid burette-tip air bubbles.
- Use the endpoint and solvent assigned to the analyte; do not transfer a factor from another salt or hydrate.
- Keep direct and back-titration signs consistent and report concordance.

## Viva Questions

### 1. Why standardize a titrant?

Its actual concentration may differ from its nominal preparation concentration.

### 2. Why perform a reagent blank?

It measures reagent or solvent consumption unrelated to the analyte.

# Assay of Phenobarbitone

Practical 11 | Medicinal Chemistry I

## Aim

To assay Phenobarbitone, and interpret the recorded observations.

## Principle

- The acidic barbiturate functionality permits the assigned alkalimetric assay. Solvent conditions affect endpoint behaviour and must follow the selected method.
- A standardized titrant reacts with the analyte according to a defined stoichiometric relationship.
- The calculation must distinguish direct titration from a blank-corrected or back-titration procedure.

## Materials and Apparatus

- Assigned phenobarbitone, standardized alkali and specified solvent/endpoint system.
- Assigned test material, standardized titrant, specified solvent and indicator or endpoint detector.
- Burette, pipette, volumetric flask, conical flask and analytical balance.

## Method

1. Identify the analyte form, the reaction and the endpoint specified for the exercise.
2. Document titrant concentration or factor and prepare a reagent blank where the method requires one.
3. Measure the prescribed test portion and dissolve or treat it by the assigned analytical method.
4. Titrate to the specified endpoint while recording initial and final burette readings.
5. Repeat the determination with independently measured portions and retain all readings.
6. Apply blank, dilution and stoichiometric corrections before reporting the result.

## Observation Record

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

| Observation / parameter | Record / finding |
|-------------------------|------------------|
| Analyte form            | _____            |
| Test mass               | _____            |
| Blank reading           | _____            |
| Test reading            | _____            |
| Method factor           | _____            |
| Assay percentage        | _____            |

## Calculations and Interpretation

- Method-corrected analyte mass = corrected titrant volume × verified analyte factor × dilution correction.
- Assay (%) = method-corrected analyte mass/test-portion mass × 100.
- Use the exact salt/hydrate and reaction factor assigned to this assay.

## Result

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

Finding: \_\_\_\_\_

Conclusion: \_\_\_\_\_

## Precautions

- Condition volumetric apparatus and avoid burette-tip air bubbles.
- Use the endpoint and solvent assigned to the analyte; do not transfer a factor from another salt or hydrate.
- Keep direct and back-titration signs consistent and report concordance.

## Viva Questions

### 1. Why standardize a titrant?

Its actual concentration may differ from its nominal preparation concentration.

### 2. Why perform a reagent blank?

It measures reagent or solvent consumption unrelated to the analyte.

# Assay of Atropine

Practical 12 | Medicinal Chemistry I

## Aim

To assay Atropine, and interpret the recorded observations.

## Principle

- Atropine is a basic alkaloid; an assigned acid-base assay must distinguish free base from a salt, hydrate and the declared reporting basis.
- A standardized titrant reacts with the analyte according to a defined stoichiometric relationship.
- The calculation must distinguish direct titration from a blank-corrected or back-titration procedure.

## Materials and Apparatus

- Prepared atropine teaching portion or completed titration data and the assigned standardized assay system.
- Assigned test material, standardized titrant, specified solvent and indicator or endpoint detector.
- Burette, pipette, volumetric flask, conical flask and analytical balance.

## Method

1. Identify the analyte form, the reaction and the endpoint specified for the exercise.
2. Document titrant concentration or factor and prepare a reagent blank where the method requires one.
3. Measure the prescribed test portion and dissolve or treat it by the assigned analytical method.
4. Titrate to the specified endpoint while recording initial and final burette readings.
5. Repeat the determination with independently measured portions and retain all readings.
6. Apply blank, dilution and stoichiometric corrections before reporting the result.

## Observation Record

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

| Observation / parameter | Record / finding |
|-------------------------|------------------|
| Analyte form            | _____            |
| Test mass               | _____            |
| Blank reading           | _____            |
| Test reading            | _____            |
| Method factor           | _____            |
| Assay percentage        | _____            |

## Calculations and Interpretation

- Method-corrected analyte mass = corrected titrant volume × verified analyte factor × dilution correction.
- Assay (%) = method-corrected analyte mass/test-portion mass × 100.
- Use the exact salt/hydrate and reaction factor assigned to this assay.

## Result

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

Finding: \_\_\_\_\_

Conclusion: \_\_\_\_\_

## Precautions

- Condition volumetric apparatus and avoid burette-tip air bubbles.
- Use the endpoint and solvent assigned to the analyte; do not transfer a factor from another salt or hydrate.
- Keep direct and back-titration signs consistent and report concordance.

## Viva Questions

### 1. Why standardize a titrant?

Its actual concentration may differ from its nominal preparation concentration.

### 2. Why perform a reagent blank?

It measures reagent or solvent consumption unrelated to the analyte.

# Assay of Ibuprofen

Practical 13 | Medicinal Chemistry I

## Aim

To assay Ibuprofen, and interpret the recorded observations.

## Principle

- The carboxylic acid group participates in neutralization. The assay must correct for blank/dilution and report the specified analyte basis.
- A standardized titrant reacts with the analyte according to a defined stoichiometric relationship.
- The calculation must distinguish direct titration from a blank-corrected or back-titration procedure.

## Materials and Apparatus

- Assigned ibuprofen, standardized alkali and the specified analytical solvent.
- Assigned test material, standardized titrant, specified solvent and indicator or endpoint detector.
- Burette, pipette, volumetric flask, conical flask and analytical balance.

## Method

1. Identify the analyte form, the reaction and the endpoint specified for the exercise.
2. Document titrant concentration or factor and prepare a reagent blank where the method requires one.
3. Measure the prescribed test portion and dissolve or treat it by the assigned analytical method.
4. Titrate to the specified endpoint while recording initial and final burette readings.
5. Repeat the determination with independently measured portions and retain all readings.
6. Apply blank, dilution and stoichiometric corrections before reporting the result.

## Observation Record

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

| Observation / parameter | Record / finding |
|-------------------------|------------------|
| Analyte form            | _____            |
| Test mass               | _____            |
| Blank reading           | _____            |
| Test reading            | _____            |
| Method factor           | _____            |
| Assay percentage        | _____            |

## Calculations and Interpretation

- Method-corrected analyte mass = corrected titrant volume × verified analyte factor × dilution correction.
- Assay (%) = method-corrected analyte mass/test-portion mass × 100.
- Use the exact salt/hydrate and reaction factor assigned to this assay.

## Result

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

Finding: \_\_\_\_\_

Conclusion: \_\_\_\_\_

## Precautions

- Condition volumetric apparatus and avoid burette-tip air bubbles.
- Use the endpoint and solvent assigned to the analyte; do not transfer a factor from another salt or hydrate.
- Keep direct and back-titration signs consistent and report concordance.

## Viva Questions

### 1. Why standardize a titrant?

Its actual concentration may differ from its nominal preparation concentration.

### 2. Why perform a reagent blank?

It measures reagent or solvent consumption unrelated to the analyte.

# Assay of Aspirin

Practical 14 | Medicinal Chemistry I

## Aim

To assay Aspirin, and interpret the recorded observations.

## Principle

- Aspirin can be assayed by an assigned hydrolysis/back-titration method. Hydrolysis changes total alkali consumption, so direct and hydrolytic factors are not interchangeable.
- A standardized titrant reacts with the analyte according to a defined stoichiometric relationship.
- The calculation must distinguish direct titration from a blank-corrected or back-titration procedure.

## Materials and Apparatus

- Assigned aspirin, standardized assay solutions and the specified hydrolysis/blank system.
- Assigned test material, standardized titrant, specified solvent and indicator or endpoint detector.
- Burette, pipette, volumetric flask, conical flask and analytical balance.

## Method

1. Identify the analyte form, the reaction and the endpoint specified for the exercise.
2. Document titrant concentration or factor and prepare a reagent blank where the method requires one.
3. Measure the prescribed test portion and dissolve or treat it by the assigned analytical method.
4. Titrate to the specified endpoint while recording initial and final burette readings.
5. Repeat the determination with independently measured portions and retain all readings.
6. Apply blank, dilution and stoichiometric corrections before reporting the result.

## Observation Record

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

| Observation / parameter | Record / finding |
|-------------------------|------------------|
| Analyte form            | _____            |
| Test mass               | _____            |
| Blank reading           | _____            |
| Test reading            | _____            |
| Method factor           | _____            |
| Assay percentage        | _____            |

## Calculations and Interpretation

- Method-corrected analyte mass = corrected titrant volume × verified analyte factor × dilution correction.
- Assay (%) = method-corrected analyte mass/test-portion mass × 100.
- Use the exact salt/hydrate and reaction factor assigned to this assay.

## Result

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

Finding: \_\_\_\_\_

Conclusion: \_\_\_\_\_

## Precautions

- Condition volumetric apparatus and avoid burette-tip air bubbles.
- Use the endpoint and solvent assigned to the analyte; do not transfer a factor from another salt or hydrate.
- Keep direct and back-titration signs consistent and report concordance.

## Viva Questions

### 1. Why standardize a titrant?

Its actual concentration may differ from its nominal preparation concentration.

### 2. Why perform a reagent blank?

It measures reagent or solvent consumption unrelated to the analyte.

# Assay of Furosemide

Practical 15 | Medicinal Chemistry I

## Aim

To assay Furosemide, and interpret the recorded observations.

## Principle

- The assigned assay measures the acidic drug under suitable solvent and endpoint conditions; water solubility alone does not define the titration medium.
- A standardized titrant reacts with the analyte according to a defined stoichiometric relationship.
- The calculation must distinguish direct titration from a blank-corrected or back-titration procedure.

## Materials and Apparatus

- Assigned furosemide, standardized assay titrant and specified solvent system.
- Assigned test material, standardized titrant, specified solvent and indicator or endpoint detector.
- Burette, pipette, volumetric flask, conical flask and analytical balance.

## Method

1. Identify the analyte form, the reaction and the endpoint specified for the exercise.
2. Document titrant concentration or factor and prepare a reagent blank where the method requires one.
3. Measure the prescribed test portion and dissolve or treat it by the assigned analytical method.
4. Titrate to the specified endpoint while recording initial and final burette readings.
5. Repeat the determination with independently measured portions and retain all readings.
6. Apply blank, dilution and stoichiometric corrections before reporting the result.

## Observation Record

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

| Observation / parameter | Record / finding |
|-------------------------|------------------|
| Analyte form            | _____            |
| Test mass               | _____            |
| Blank reading           | _____            |
| Test reading            | _____            |
| Method factor           | _____            |
| Assay percentage        | _____            |

## Calculations and Interpretation

- Method-corrected analyte mass = corrected titrant volume × verified analyte factor × dilution correction.
- Assay (%) = method-corrected analyte mass/test-portion mass × 100.
- Use the exact salt/hydrate and reaction factor assigned to this assay.

## Result

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

Finding: \_\_\_\_\_

Conclusion: \_\_\_\_\_

## Precautions

- Condition volumetric apparatus and avoid burette-tip air bubbles.
- Use the endpoint and solvent assigned to the analyte; do not transfer a factor from another salt or hydrate.
- Keep direct and back-titration signs consistent and report concordance.

## Viva Questions

### 1. Why standardize a titrant?

Its actual concentration may differ from its nominal preparation concentration.

### 2. Why perform a reagent blank?

It measures reagent or solvent consumption unrelated to the analyte.

# Partition Coefficient of Two Assigned Drugs

Practical 16 | Medicinal Chemistry I

## Aim

To study Partition Coefficient of Two Assigned Drugs, and interpret the recorded observations.

## Principle

- Compare distribution in two immiscible phases at a stated pH and temperature. Intrinsic neutral-species partitioning differs from total-species distribution when a drug ionizes.
- 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

- Two instructor-selected drugs, prepared equilibrium phase solutions and assigned concentration assays.
- Assigned test material and calibration or comparison material.
- The apparatus specified for this property, a balance or volumetric equipment where relevant, and a record sheet.

## Method

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

## Observation Record

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

| Observation / parameter        | Record / finding |
|--------------------------------|------------------|
| Drug                           | _____            |
| Organic/aqueous concentrations | _____            |
| pH                             | _____            |
| Temperature                    | _____            |
| Replicate ratios               | _____            |

## Calculations and Interpretation

- $D = \text{total organic-phase concentration} / \text{total aqueous-phase concentration}$ .
- For neutral-species partitioning,  $P$  uses the same molecular species in both phases;  $\log P = \log_{10}(P)$ .
- Compare the two drugs only under equivalent phase and measurement conditions.

## Result

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

Finding: \_\_\_\_\_

Conclusion: \_\_\_\_\_

## Precautions

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

## Viva Questions

### 1. Why control the test conditions?

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

### 2. Why retain replicate readings?

They show repeatability and help identify an anomalous measurement.

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