

# Continue Learning with FirstHope

## Complete Notes on the FirstHope App

**Complete subject and semesters are available on FirstHope app only.**

- Get complete subject and semester notes in the FirstHope app.
- Available for B.Pharm, D.Pharm, M.Pharm and Pharm.D students.

**[Click here to get the FirstHope app](#)**

## Join the FirstHope Telegram Channel

Join FirstHope Notes and keep up with channel updates.

**[Click here to join Telegram](#)**

## Instagram

@firsthope.official

**[Click here to follow Instagram](#)**

## YouTube Channel

FirstHope-Pharmacy / Live streams

**[Click here to watch the YouTube channel](#)**

## WhatsApp Channel

Follow FirstHope on WhatsApp.

**[Click here to join the WhatsApp channel](#)**

## FirstHope Website

firsthopenotes.com

**[Click here to visit the website](#)**

# Semester 2 - Practical Notes

## Pharmaceutical Organic Chemistry

### Complete Subject Manual

16 practical files and activities. Use the PDF bookmarks to navigate. Page numbers restart within each practical; content page counts below exclude promotion pages.

### Contents

| No. | Practical                                      | Content pages |
|-----|------------------------------------------------|---------------|
| 1   | Preliminary Examination of Organic Compounds   | 2             |
| 2   | Detection of Nitrogen Sulphur and Halogens     | 2             |
| 3   | Functional Group Identification Tests          | 3             |
| 4   | Derivatives and Physical-Constant Confirmation | 2             |
| 5   | Systematic Analysis of a Carboxylic Acid       | 2             |
| 6   | Systematic Analysis of a Phenol                | 2             |
| 7   | Systematic Analysis of an Alcohol              | 2             |
| 8   | Systematic Analysis of an Aldehyde             | 2             |
| 9   | Systematic Analysis of an Ester                | 2             |
| 10  | Systematic Analysis of an Amide                | 2             |
| 11  | Systematic Analysis of an Aromatic Hydrocarbon | 2             |
| 12  | Systematic Analysis of an Amino Ester          | 2             |
| 13  | Building Ball-and-Stick Molecular Models       | 2             |
| 14  | Recrystallisation of Benzoic Acid              | 2             |
| 15  | Recrystallisation of Acetanilide               | 2             |
| 16  | Recrystallisation of Naphthalene               | 2             |

# Preliminary Examination of Organic Compounds

Experiment 1 - Practical Notes

Name: \_\_\_\_\_ Roll no.: \_\_\_\_\_ Date: \_\_\_\_\_

## Aim

To record physical appearance, solubility and selected screening reactions before systematic identification.

## Principle

Physical state and solubility narrow the possible chemical classes. Acid-base conversion may turn a neutral organic compound into a water-soluble ionic form. Aromaticity and unsaturation cannot be established reliably from appearance or a sooty flame alone.

## Requirements

- Instructor-assigned unknowns, water, prescribed dilute acid/base and bicarbonate reagents.
- Small labelled tubes, pipettes, pH paper and instructor-approved unsaturation-test reagents.

## Procedure

### 1. Record appearance

Observe state and colour without direct smelling or tasting. Odour descriptions are taken from an approved sealed demonstration or the supplied data sheet.

### 2. Water test

Use the prescribed fixed quantity and water volume. Record dissolved, partly dissolved, insoluble or two phases; temperature and quantity must be stated.

### 3. Acid-base solubility

On new portions test the assigned dilute hydroxide, bicarbonate and acid systems. Solubility in hydroxide but not bicarbonate may support a weak acid such as a phenol; solubility in dilute acid may support a basic amine.

### 4. Unsaturation screen

The instructor demonstrates the prescribed dilute permanganate or bromine test with controls. Decolourisation can also result from oxidation of other reactive groups, so retain the limitation.

### 5. Aromaticity assessment

Combine the assigned spectroscopic or structural evidence with the later chemical pattern. Do not equate dark burning with definitive aromaticity.

### 6. Plan tests

Use the solubility evidence to select elemental and functional-group checks on fresh material; keep an untouched portion for physical-constant confirmation.

**Pharmaceutical Organic Chemistry**

---

**Observations and Record**

| Test                              | Observation | Tentative inference |
|-----------------------------------|-------------|---------------------|
| State / colour / supplied odour   | _____       | _____               |
| Water / dilute acid / hydroxide   | _____       | _____               |
| Bicarbonate / unsaturation screen | _____       | _____               |

**Worked Example**

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

- A solid dissolving in bicarbonate with gas evolution supports a carboxylic-acid candidate, but confirmation still needs the rest of the analysis.

**Result**

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

**Precautions**

- Use instructor-assigned quantities and prepared test reagents in the designated laboratory; wear eye protection and use the required fume hood.
- Do not taste unknowns, smell containers directly, burn unknown halogenated material or heat sealed vessels.
- Use fresh separate portions for independent tests. A single colour reaction does not establish molecular identity.

**Viva Questions and Answers****1. Why record the quantity in a solubility test?**

A limited-solubility material may dissolve at a small loading but form a separate phase at a higher loading.

**2. Does permanganate decolourisation prove an alkene?**

No. Other oxidisable substances may react.

**3. Why retain an untouched portion?**

It is needed for independent confirmation and avoids contamination from earlier tests.

# Detection of Nitrogen Sulphur and Halogens

Experiment 2 - Practical Notes

Name: \_\_\_\_\_ Roll no.: \_\_\_\_\_ Date: \_\_\_\_\_

## Aim

To interpret Lassaigne elemental tests using instructor-prepared fusion extracts and appropriate controls.

## Principle

Sodium-fusion analysis converts suitable covalently bound elements into water-soluble ionic forms. Nitrogen is detected through the iron-cyanide colour sequence, sulphur through a sulphide reaction and halogens through silver-halide formation after the prescribed removal of interfering ions.

## Requirements

- Teacher-prepared and labelled Lassaigne extracts, positive and negative controls.
- Prepared ferrous reagent, nitroprusside reagent, silver nitrate and instructor-treated extract for the halogen branch.

## Procedure

### 1. Obtain the extract

Record the teacher-prepared extract identifier. Hot fusion, sodium handling and extract treatment are performed only by trained staff in the designated setup.

### 2. Nitrogen branch

Use a separate extract portion for the instructor's iron-reagent sequence. Observe the Prussian-blue result against known positive and negative controls; record an inconclusive or failed-control result as such.

### 3. Sulphur branch

On a new portion add the assigned fresh nitroprusside reagent. A violet/purple response supports sulphide in the prepared extract; observe promptly as colour can fade.

### 4. Halogen branch

Use only the extract portion prepared by the instructor for this branch. Add the specified silver nitrate reagent and observe white, cream or yellow silver-halide precipitate. Further prescribed solubility comparison can help distinguish halides.

### 5. Consider combinations

When both nitrogen and sulphur occur, the fusion chemistry may produce a thiocyanate response. Interpret with the teacher's method; an unsuitable fusion can give a false negative.

### 6. Record elements

List detected elements separately from functional-group conclusions. A nitrogen-positive result alone does not distinguish an amine from an amide or nitro compound.

**Pharmaceutical Organic Chemistry**

---

**Observations and Record**

| Branch   | Supporting observation             | Record / limitation |
|----------|------------------------------------|---------------------|
| Nitrogen | Prussian-blue response             | _____               |
| Sulphur  | Violet nitroprusside response      | _____               |
| Halogens | AgCl white; AgBr cream; AgI yellow | _____               |

**Worked Example**

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

- Positive nitrogen with negative sulphur/halogen narrows the classes but does not identify a specific molecule. Combine it with the functional tests and physical constant.

**Result**

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

**Precautions**

- Do not prepare or acidify sodium-fusion extracts independently; some treatments can release hazardous vapours.
- Silver-containing residues are disposed of through the prescribed waste system; do not store reactive test mixtures.

**Viva Questions and Answers****1. Why use fusion before these tests?**

The original covalent forms are not generally detected by ordinary ionic reagents.

**2. Why remove halogen-test interference?**

Other fusion ions may form misleading silver precipitates.

**3. Does a negative nitrogen test always prove absence?**

No. Extract preparation, sensitivity and controls must be satisfactory.

# Functional Group Identification Tests

Experiment 3 - Practical Notes

Name: \_\_\_\_\_ Roll no.: \_\_\_\_\_ Date: \_\_\_\_\_

## Aim

To select and interpret tests for the eleven functional classes listed in the syllabus.

## Principle

Functional-group reactions provide class evidence. Identification requires a coherent pattern, appropriate controls and an independent physical or derivative confirmation; no universal colour test identifies every member of a class.

## Requirements

- Teacher-assigned unknowns and known comparison compounds; clean tubes and separate portions.
- Prepared reagents for the selected tests, laboratory method sheet and prescribed fume-hood setup.

## Procedure

### 1. Select a branch

Use appearance, solubility and elemental results to choose the relevant functional-group checks.

### 2. Run controls

Perform the chosen test on a known positive and the appropriate negative/reagent blank under the same conditions.

### 3. Test fresh portions

Follow the institution's reagent order, quantity and heating conditions; do not combine successive test mixtures.

### 4. Record the observation

Describe the colour, precipitate, gas or solubility before writing an inference. Note timing and any failure of the control.

### 5. Confirm

Select a second independent check and physical-constant/derivative evidence before assigning the candidate.

## Test Guide

### Carboxylic acids

- Test: Bicarbonate reaction.
- Supporting observation: Gas evolution and dissolution support formation of a carboxylate.
- Interpretation limit: An acid-base result is class evidence; confirm with physical data and an appropriate derivative.

## Pharmaceutical Organic Chemistry

---

### Phenols

- Test: Ferric-chloride complex on a suitable solution.
- Supporting observation: Violet, blue, green or other characteristic complex may develop.
- Interpretation limit: Enols and other complexing substances can interfere; not every phenol gives the same colour.

### Amides

- Test: Instructor-controlled alkaline hydrolysis and product checks.
- Supporting observation: Suitable amides yield a carboxylate and an ammonia/amine-related product.
- Interpretation limit: Do not sniff evolved gas; use the assigned indicator/product test and a control.

### Amines

- Test: Acid solubility and selected Hinsberg or aromatic-amine test.
- Supporting observation: Salt formation supports basicity. Hinsberg behaviour can help distinguish some primary, secondary and tertiary amines.
- Interpretation limit: Not all amines have identical solubility or reactivity; aromatic-primary tests do not apply to every amine.

### Aldehydes

- Test: Prepared 2,4-DNP followed by fresh instructor-led Tollens.
- Supporting observation: Carbonyl derivative forms; an appropriate aldehyde may reduce the silver reagent.
- Interpretation limit: Tollens is prepared fresh and residues are removed immediately by the instructor; do not store it.

### Ketones

- Test: Prepared 2,4-DNP with aldehyde-discrimination tests.
- Supporting observation: A hydrazone precipitate supports a reactive carbonyl; ordinary ketones are usually Tollens-negative.
- Interpretation limit: Alpha-hydroxy ketones and other reducing substances may give exceptions.

### Alcohols

- Test: Assigned ceric reagent and, where appropriate, Lucas comparison.
- Supporting observation: A reagent-specific colour response supports alcohol; substitution-rate comparison is useful for selected aliphatic alcohols.
- Interpretation limit: Benzylic/allylic primary alcohols may react rapidly; Lucas timing is not a universal classification.

### Esters

- Test: Hydroxamic-acid formation followed by ferric reagent.
- Supporting observation: A red/violet complex after the prescribed reaction can support an ester.
- Interpretation limit: Use reagent blanks; coloured starting material and other reactive groups can interfere.

## Pharmaceutical Organic Chemistry

---

### Aromatic hydrocarbons

- Test: Solubility, supplied spectrum and physical constant.
- Supporting observation: A neutral, non-polar pattern plus aromatic spectral evidence supports this class.
- Interpretation limit: A sooty flame is supporting at most and is not a safe universal test for unknowns.

### Halogenated hydrocarbons

- Test: Prepared fusion-extract halogen test; supplied structural evidence.
- Supporting observation: A silver-halide reaction supports halogen in the compound.
- Interpretation limit: The elemental result does not identify the carbon skeleton or establish the halogen position.

### Nitro compounds

- Test: Instructor-demonstrated reduction followed by amine evidence.
- Supporting observation: A changed response after the assigned reduction can support a nitro group.
- Interpretation limit: Nitrogen detection alone is insufficient; other reducible groups and reduction failure must be considered.

### Observations and Record

| Test / control | Observed result | Class inference / limitation |
|----------------|-----------------|------------------------------|
| _____          | _____           | _____                        |
| _____          | _____           | _____                        |

### Result

State the functional class supported by the complete pattern and the independent confirmation used.

### Precautions

- Use instructor-assigned quantities and prepared test reagents in the designated laboratory; wear eye protection and use the required fume hood.
- Do not taste unknowns, smell containers directly, burn unknown halogenated material or heat sealed vessels.
- Use fresh separate portions for independent tests. A single colour reaction does not establish molecular identity.
- Use commercially prepared wet 2,4-DNP reagent; do not dry or isolate the reagent. Diazonium, silver-reagent and reduction demonstrations follow the instructor's controlled method.

### Viva Questions and Answers

#### 1. Does 2,4-DNP distinguish aldehydes from ketones?

No. It supports a reactive carbonyl and requires additional discrimination.

#### 2. Why test a reagent blank?

It shows whether the reagent itself contributes colour or precipitate.

#### 3. Why use several kinds of evidence?

Class tests and physical constants can overlap, while their combined pattern is more informative.

# Derivatives and Physical-Constant Confirmation

Experiment 4 - Practical Notes

Name: \_\_\_\_\_ Roll no.: \_\_\_\_\_ Date: \_\_\_\_\_

## Aim

To prepare an appropriate assigned derivative and confirm a candidate with its measured melting or boiling behaviour.

## Principle

A derivative converts a functional group into a recognisable product with useful physical properties. Purity, heating rate, pressure and polymorphism can affect a physical constant, so a matching value supports identity only when the chemical evidence is also consistent.

## Requirements

- Assigned compound, teacher-selected derivative reaction and prepared reagents.
- Suitable purification apparatus, dry capillary or approved boiling-point setup, calibrated instrument and authentic comparison where available.

## Procedure

### 1. Choose the derivative

Use the candidate functional group to select the laboratory reaction: carbonyl hydrazone, assigned ester/amide, phenol ester or another validated derivative. Not every unknown needs the same reagent.

### 2. React under the assigned method

Follow the teacher-approved microscale quantities, temperature and reaction interval. Record the actual reagents and amounts rather than using an unrelated recipe.

### 3. Isolate and purify

Collect the product using the prescribed filtration/extraction, then wash and recrystallise if appropriate. Dry it to the defined condition before thermal measurement.

### 4. Measure melting range

Pack a short even column of dry solid in the capillary. Use the instrument's approved heating programme, reducing the rate near the expected range; record onset and complete melting.

### 5. Measure boiling behaviour

For a liquid, use the approved micro boiling-point or distillation setup operated by the instructor. Record atmospheric pressure and do not heat a closed system.

### 6. Confirm with controls

Compare the original and derivative physical data with the teacher's verified values. If appropriate, an authentic mixed-melting comparison should remain narrow rather than show a substantial depression.

**Pharmaceutical Organic Chemistry**

---

**Observations and Record**

| Record                                                  | Entry |
|---------------------------------------------------------|-------|
| Candidate / derivative name / reaction                  | _____ |
| Amounts / isolated dry mass                             | _____ |
| Melting onset / clear point or boiling point / pressure | _____ |
| Authentic comparison / conclusion                       | _____ |

**Worked Example**

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

- If theoretical derivative mass is 0.500 g and dry isolated mass is 0.350 g, isolated yield =  $0.350/0.500 \times 100 = 70.0\%$ . Purity is not established by yield alone.

**Result**

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

**Precautions**

- Use instructor-assigned quantities and prepared test reagents in the designated laboratory; wear eye protection and use the required fume hood.
- Do not taste unknowns, smell containers directly, burn unknown halogenated material or heat sealed vessels.
- Use fresh separate portions for independent tests. A single colour reaction does not establish molecular identity.

**Viva Questions and Answers****1. Why dry the solid?**

Residual solvent may broaden or depress the melting range.

**2. Why heat slowly near melting?**

It reduces thermal lag and improves the recorded onset/clear point.

**3. Does an unchanged mixed-melting range prove identity by itself?**

It supports identity, but the full chemical and physical pattern is still required.

# Systematic Analysis of a Carboxylic Acid

Experiment 5 - Practical Notes

Name: \_\_\_\_\_ Roll no.: \_\_\_\_\_ Date: \_\_\_\_\_

## Aim

To organise an illustrative unknown-identification record and show how the assigned candidate is supported.

## Principle

This is a worked teaching case, not an observation to copy into a laboratory record. An actual unknown is assigned by the instructor and may give different findings. A candidate is supported by consistent preliminary, elemental, functional and physical evidence.

## Requirements

- Instructor-assigned unknown and the corresponding known control; the method notes in this subject.
- Prepared test reagents, separate test portions and calibrated physical-constant apparatus.

## Procedure

### 1. Preliminary examination

Record the actual state and water behaviour at the prescribed quantity and temperature. Compare with the illustrative pattern below.

### 2. Solubility classification

Test fresh portions in the assigned acid/base reagents. Describe dissolution or effervescence before inferring a class.

### 3. Elements

Use the instructor-prepared extract or provided elemental report. Distinguish an element finding from a functional-group conclusion.

### 4. Functional confirmation

Bicarbonate reaction supports a carboxylic acid; confirm a coherent acid pattern

### 5. Physical confirmation

Measure the dried solid melting range and compare with verified benzoic acid; perform the assigned derivative or mixed-melting comparison.

### 6. Conclude

Write the candidate only after reviewing all evidence; list a plausible alternative and the test that distinguishes it.

**Pharmaceutical Organic Chemistry****Observations and Record**

| Evidence                | Illustrative finding                                                             |
|-------------------------|----------------------------------------------------------------------------------|
| State / water behaviour | White crystalline solid; Low water solubility at room temperature                |
| Acid-base pattern       | Dissolves in bicarbonate with carbon-dioxide evolution and in hydroxide          |
| Elements                | Nitrogen, sulphur and halogens negative in the teaching pattern                  |
| Functional pattern      | Bicarbonate reaction supports a carboxylic acid; confirm a coherent acid pattern |
| Physical constant       | Typical melting region about 121-123°C, dependent on purity and method           |
| Supported candidate     | Benzoic acid                                                                     |

**Worked Example**

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

- Record the candidate as Benzoic acid only if your own results support the pattern. An anomalous physical constant should be investigated before final assignment.

**Result**

Complete the actual unknown record with its identification evidence and justified candidate.

**Precautions**

- Use instructor-assigned quantities and prepared test reagents in the designated laboratory; wear eye protection and use the required fume hood.
- Do not taste unknowns, smell containers directly, burn unknown halogenated material or heat sealed vessels.
- Use fresh separate portions for independent tests. A single colour reaction does not establish molecular identity.
- Phenols may dissolve in hydroxide but usually do not show the same bicarbonate response.

**Viva Questions and Answers****1. Can the title identify an actual unknown?**

No. The title describes this worked case; the actual material must be analysed.

**2. Why perform a confirmatory measurement?**

Different molecules can share solubility and functional-group reactions.

**3. What belongs in the result?**

The observed evidence, proposed identity and any unresolved limitation.

# Systematic Analysis of a Phenol

Experiment 6 - Practical Notes

Name: \_\_\_\_\_ Roll no.: \_\_\_\_\_ Date: \_\_\_\_\_

## Aim

To organise an illustrative unknown-identification record and show how the assigned candidate is supported.

## Principle

This is a worked teaching case, not an observation to copy into a laboratory record. An actual unknown is assigned by the instructor and may give different findings. A candidate is supported by consistent preliminary, elemental, functional and physical evidence.

## Requirements

- Instructor-assigned unknown and the corresponding known control; the method notes in this subject.
- Prepared test reagents, separate test portions and calibrated physical-constant apparatus.

## Procedure

### 1. Preliminary examination

Record the actual state and water behaviour at the prescribed quantity and temperature. Compare with the illustrative pattern below.

### 2. Solubility classification

Test fresh portions in the assigned acid/base reagents. Describe dissolution or effervescence before inferring a class.

### 3. Elements

Use the instructor-prepared extract or provided elemental report. Distinguish an element finding from a functional-group conclusion.

### 4. Functional confirmation

Run the assigned ferric-complex and phenol confirmation with a known control; interpret colour under that method

### 5. Physical confirmation

Measure the dry parent solid and the assigned purified phenol derivative, using verified comparison values.

### 6. Conclude

Write the candidate only after reviewing all evidence; list a plausible alternative and the test that distinguishes it.

**Pharmaceutical Organic Chemistry****Observations and Record**

| Evidence                | Illustrative finding                                                                                             |
|-------------------------|------------------------------------------------------------------------------------------------------------------|
| State / water behaviour | White to pale solid; Poor water solubility                                                                       |
| Acid-base pattern       | Dissolves in hydroxide; ordinary bicarbonate response is negative                                                |
| Elements                | Nitrogen, sulphur and halogens negative in the teaching pattern                                                  |
| Functional pattern      | Run the assigned ferric-complex and phenol confirmation with a known control; interpret colour under that method |
| Physical constant       | Typical melting region near 121°C                                                                                |
| Supported candidate     | 2-Naphthol                                                                                                       |

**Worked Example**

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

- Record the candidate as 2-Naphthol only if your own results support the pattern. An anomalous physical constant should be investigated before final assignment.

**Result**

Complete the actual unknown record with its identification evidence and justified candidate.

**Precautions**

- Use instructor-assigned quantities and prepared test reagents in the designated laboratory; wear eye protection and use the required fume hood.
- Do not taste unknowns, smell containers directly, burn unknown halogenated material or heat sealed vessels.
- Use fresh separate portions for independent tests. A single colour reaction does not establish molecular identity.
- Its melting region overlaps benzoic acid; melting point alone cannot distinguish them.

**Viva Questions and Answers****1. Can the title identify an actual unknown?**

No. The title describes this worked case; the actual material must be analysed.

**2. Why perform a confirmatory measurement?**

Different molecules can share solubility and functional-group reactions.

**3. What belongs in the result?**

The observed evidence, proposed identity and any unresolved limitation.

# Systematic Analysis of an Alcohol

Experiment 7 - Practical Notes

Name: \_\_\_\_\_ Roll no.: \_\_\_\_\_ Date: \_\_\_\_\_

## Aim

To organise an illustrative unknown-identification record and show how the assigned candidate is supported.

## Principle

This is a worked teaching case, not an observation to copy into a laboratory record. An actual unknown is assigned by the instructor and may give different findings. A candidate is supported by consistent preliminary, elemental, functional and physical evidence.

## Requirements

- Instructor-assigned unknown and the corresponding known control; the method notes in this subject.
- Prepared test reagents, separate test portions and calibrated physical-constant apparatus.

## Procedure

### 1. Preliminary examination

Record the actual state and water behaviour at the prescribed quantity and temperature. Compare with the illustrative pattern below.

### 2. Solubility classification

Test fresh portions in the assigned acid/base reagents. Describe dissolution or effervescence before inferring a class.

### 3. Elements

Use the instructor-prepared extract or provided elemental report. Distinguish an element finding from a functional-group conclusion.

### 4. Functional confirmation

The assigned alcohol reaction supports OH; carbonyl test on fresh unoxidised material is negative

### 5. Physical confirmation

Use the instructor's boiling-point data and a suitable assigned alcohol derivative or supplied spectrum.

### 6. Conclude

Write the candidate only after reviewing all evidence; list a plausible alternative and the test that distinguishes it.

## Observations and Record

| Evidence                | Illustrative finding                                                                              |
|-------------------------|---------------------------------------------------------------------------------------------------|
| State / water behaviour | Colourless liquid; Limited miscibility; excess material can form a separate phase                 |
| Acid-base pattern       | No ordinary bicarbonate effervescence; not a carboxylic acid                                      |
| Elements                | Nitrogen, sulphur and halogens negative in the teaching pattern                                   |
| Functional pattern      | The assigned alcohol reaction supports OH; carbonyl test on fresh unoxidised material is negative |
| Physical constant       | Boiling region approximately 203-205°C at near-atmospheric pressure                               |
| Supported candidate     | Benzyl alcohol                                                                                    |

## Worked Example

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

- Record the candidate as Benzyl alcohol only if your own results support the pattern. An anomalous physical constant should be investigated before final assignment.

## Result

Complete the actual unknown record with its identification evidence and justified candidate.

## Precautions

- Use instructor-assigned quantities and prepared test reagents in the designated laboratory; wear eye protection and use the required fume hood.
- Do not taste unknowns, smell containers directly, burn unknown halogenated material or heat sealed vessels.
- Use fresh separate portions for independent tests. A single colour reaction does not establish molecular identity.
- A primary benzylic alcohol can react rapidly in Lucas conditions; do not misclassify it as tertiary from timing alone.

## Viva Questions and Answers

### 1. Can the title identify an actual unknown?

No. The title describes this worked case; the actual material must be analysed.

### 2. Why perform a confirmatory measurement?

Different molecules can share solubility and functional-group reactions.

### 3. What belongs in the result?

The observed evidence, proposed identity and any unresolved limitation.

# Systematic Analysis of an Aldehyde

Experiment 8 - Practical Notes

Name: \_\_\_\_\_ Roll no.: \_\_\_\_\_ Date: \_\_\_\_\_

## Aim

To organise an illustrative unknown-identification record and show how the assigned candidate is supported.

## Principle

This is a worked teaching case, not an observation to copy into a laboratory record. An actual unknown is assigned by the instructor and may give different findings. A candidate is supported by consistent preliminary, elemental, functional and physical evidence.

## Requirements

- Instructor-assigned unknown and the corresponding known control; the method notes in this subject.
- Prepared test reagents, separate test portions and calibrated physical-constant apparatus.

## Procedure

### 1. Preliminary examination

Record the actual state and water behaviour at the prescribed quantity and temperature. Compare with the illustrative pattern below.

### 2. Solubility classification

Test fresh portions in the assigned acid/base reagents. Describe dissolution or effervescence before inferring a class.

### 3. Elements

Use the instructor-prepared extract or provided elemental report. Distinguish an element finding from a functional-group conclusion.

### 4. Functional confirmation

Prepared 2,4-DNP gives carbonyl evidence; fresh instructor-led Tollens supports an aldehyde

### 5. Physical confirmation

Prepare the assigned carbonyl derivative with ready-to-use wet reagent; purify and measure its melting range against the verified derivative value.

### 6. Conclude

Write the candidate only after reviewing all evidence; list a plausible alternative and the test that distinguishes it.

**Pharmaceutical Organic Chemistry****Observations and Record**

| Evidence                | Illustrative finding                                                                        |
|-------------------------|---------------------------------------------------------------------------------------------|
| State / water behaviour | Colourless to pale liquid; Poor water solubility / separate phase at the test loading       |
| Acid-base pattern       | No ordinary carboxylic-acid bicarbonate pattern in fresh material                           |
| Elements                | Nitrogen, sulphur and halogens negative in the teaching pattern                             |
| Functional pattern      | Prepared 2,4-DNP gives carbonyl evidence; fresh instructor-led Tollens supports an aldehyde |
| Physical constant       | Boiling region near 178-179°C at near-atmospheric pressure                                  |
| Supported candidate     | Benzaldehyde                                                                                |

**Worked Example**

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

- Record the candidate as Benzaldehyde only if your own results support the pattern. An anomalous physical constant should be investigated before final assignment.

**Result**

Complete the actual unknown record with its identification evidence and justified candidate.

**Precautions**

- Use instructor-assigned quantities and prepared test reagents in the designated laboratory; wear eye protection and use the required fume hood.
- Do not taste unknowns, smell containers directly, burn unknown halogenated material or heat sealed vessels.
- Use fresh separate portions for independent tests. A single colour reaction does not establish molecular identity.
- Oxidation during storage can introduce benzoic acid; check the actual batch and do not infer identity from odour.

**Viva Questions and Answers****1. Can the title identify an actual unknown?**

No. The title describes this worked case; the actual material must be analysed.

**2. Why perform a confirmatory measurement?**

Different molecules can share solubility and functional-group reactions.

**3. What belongs in the result?**

The observed evidence, proposed identity and any unresolved limitation.

# Systematic Analysis of an Ester

Experiment 9 - Practical Notes

Name: \_\_\_\_\_ Roll no.: \_\_\_\_\_ Date: \_\_\_\_\_

## Aim

To organise an illustrative unknown-identification record and show how the assigned candidate is supported.

## Principle

This is a worked teaching case, not an observation to copy into a laboratory record. An actual unknown is assigned by the instructor and may give different findings. A candidate is supported by consistent preliminary, elemental, functional and physical evidence.

## Requirements

- Instructor-assigned unknown and the corresponding known control; the method notes in this subject.
- Prepared test reagents, separate test portions and calibrated physical-constant apparatus.

## Procedure

### 1. Preliminary examination

Record the actual state and water behaviour at the prescribed quantity and temperature. Compare with the illustrative pattern below.

### 2. Solubility classification

Test fresh portions in the assigned acid/base reagents. Describe dissolution or effervescence before inferring a class.

### 3. Elements

Use the instructor-prepared extract or provided elemental report. Distinguish an element finding from a functional-group conclusion.

### 4. Functional confirmation

Assigned hydroxamic-acid/ferric reaction supports an ester. Controlled hydrolysis can produce a benzoate/benzoic-acid fraction

### 5. Physical confirmation

Use approved boiling-point data and identify the assigned hydrolysis product after purification.

### 6. Conclude

Write the candidate only after reviewing all evidence; list a plausible alternative and the test that distinguishes it.

**Pharmaceutical Organic Chemistry****Observations and Record**

| Evidence                | Illustrative finding                                                                                                           |
|-------------------------|--------------------------------------------------------------------------------------------------------------------------------|
| State / water behaviour | Colourless liquid; Poor water solubility / immiscible at ordinary test loading                                                 |
| Acid-base pattern       | Neutral preliminary pattern; hydrolysis changes the products                                                                   |
| Elements                | Nitrogen, sulphur and halogens negative in the teaching pattern                                                                |
| Functional pattern      | Assigned hydroxamic-acid/ferric reaction supports an ester. Controlled hydrolysis can produce a benzoate/benzoic-acid fraction |
| Physical constant       | Boiling region near 212°C at near-atmospheric pressure                                                                         |
| Supported candidate     | Ethyl benzoate                                                                                                                 |

**Worked Example**

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

- Record the candidate as Ethyl benzoate only if your own results support the pattern. An anomalous physical constant should be investigated before final assignment.

**Result**

Complete the actual unknown record with its identification evidence and justified candidate.

**Precautions**

- Use instructor-assigned quantities and prepared test reagents in the designated laboratory; wear eye protection and use the required fume hood.
- Do not taste unknowns, smell containers directly, burn unknown halogenated material or heat sealed vessels.
- Use fresh separate portions for independent tests. A single colour reaction does not establish molecular identity.
- Hydrolysis requires the specific supervised laboratory method; unreacted ester is not equivalent to the isolated acid product.

**Viva Questions and Answers****1. Can the title identify an actual unknown?**

No. The title describes this worked case; the actual material must be analysed.

**2. Why perform a confirmatory measurement?**

Different molecules can share solubility and functional-group reactions.

**3. What belongs in the result?**

The observed evidence, proposed identity and any unresolved limitation.

# Systematic Analysis of an Amide

Experiment 10 - Practical Notes

Name: \_\_\_\_\_ Roll no.: \_\_\_\_\_ Date: \_\_\_\_\_

## Aim

To organise an illustrative unknown-identification record and show how the assigned candidate is supported.

## Principle

This is a worked teaching case, not an observation to copy into a laboratory record. An actual unknown is assigned by the instructor and may give different findings. A candidate is supported by consistent preliminary, elemental, functional and physical evidence.

## Requirements

- Instructor-assigned unknown and the corresponding known control; the method notes in this subject.
- Prepared test reagents, separate test portions and calibrated physical-constant apparatus.

## Procedure

### 1. Preliminary examination

Record the actual state and water behaviour at the prescribed quantity and temperature. Compare with the illustrative pattern below.

### 2. Solubility classification

Test fresh portions in the assigned acid/base reagents. Describe dissolution or effervescence before inferring a class.

### 3. Elements

Use the instructor-prepared extract or provided elemental report. Distinguish an element finding from a functional-group conclusion.

### 4. Functional confirmation

Instructor-controlled amide hydrolysis gives the corresponding acid/amine products; the original compound is not a free primary amine

### 5. Physical confirmation

Purify the parent by hot-water recrystallisation and compare the measured range with the assigned authentic material; use the prescribed hydrolysis-product confirmation.

### 6. Conclude

Write the candidate only after reviewing all evidence; list a plausible alternative and the test that distinguishes it.

**Pharmaceutical Organic Chemistry****Observations and Record**

| Evidence                | Illustrative finding                                                                                                                  |
|-------------------------|---------------------------------------------------------------------------------------------------------------------------------------|
| State / water behaviour | White crystalline solid; Poor cold-water solubility                                                                                   |
| Acid-base pattern       | Neutral preliminary pattern; does not behave as a simple carboxylic acid                                                              |
| Elements                | Nitrogen positive; sulphur and halogens negative in the teaching pattern                                                              |
| Functional pattern      | Instructor-controlled amide hydrolysis gives the corresponding acid/amine products; the original compound is not a free primary amine |
| Physical constant       | Typical melting region about 113-116°C, with certified standards having batch-specific values                                         |
| Supported candidate     | Acetanilide                                                                                                                           |

**Worked Example**

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

- Record the candidate as Acetanilide only if your own results support the pattern. An anomalous physical constant should be investigated before final assignment.

**Result**

Complete the actual unknown record with its identification evidence and justified candidate.

**Precautions**

- Use instructor-assigned quantities and prepared test reagents in the designated laboratory; wear eye protection and use the required fume hood.
- Do not taste unknowns, smell containers directly, burn unknown halogenated material or heat sealed vessels.
- Use fresh separate portions for independent tests. A single colour reaction does not establish molecular identity.
- Do not mistake nitrogen detection alone for proof of an amide.

**Viva Questions and Answers****1. Can the title identify an actual unknown?**

No. The title describes this worked case; the actual material must be analysed.

**2. Why perform a confirmatory measurement?**

Different molecules can share solubility and functional-group reactions.

**3. What belongs in the result?**

The observed evidence, proposed identity and any unresolved limitation.

# Systematic Analysis of an Aromatic Hydrocarbon

Experiment 11 - Practical Notes

Name: \_\_\_\_\_ Roll no.: \_\_\_\_\_ Date: \_\_\_\_\_

## Aim

To organise an illustrative unknown-identification record and show how the assigned candidate is supported.

## Principle

This is a worked teaching case, not an observation to copy into a laboratory record. An actual unknown is assigned by the instructor and may give different findings. A candidate is supported by consistent preliminary, elemental, functional and physical evidence.

## Requirements

- Instructor-assigned unknown and the corresponding known control; the method notes in this subject.
- Prepared test reagents, separate test portions and calibrated physical-constant apparatus.

## Procedure

### 1. Preliminary examination

Record the actual state and water behaviour at the prescribed quantity and temperature. Compare with the illustrative pattern below.

### 2. Solubility classification

Test fresh portions in the assigned acid/base reagents. Describe dissolution or effervescence before inferring a class.

### 3. Elements

Use the instructor-prepared extract or provided elemental report. Distinguish an element finding from a functional-group conclusion.

### 4. Functional confirmation

Functional screens exclude the tested acid, phenol and carbonyl classes; supplied spectral evidence supports an aromatic hydrocarbon

### 5. Physical confirmation

Measure the purified parent and compare with an authentic mixed-melting check or supplied spectrum; no hazardous derivative is required merely to force a precipitate.

### 6. Conclude

Write the candidate only after reviewing all evidence; list a plausible alternative and the test that distinguishes it.

**Pharmaceutical Organic Chemistry****Observations and Record**

| Evidence                | Illustrative finding                                                                                                                 |
|-------------------------|--------------------------------------------------------------------------------------------------------------------------------------|
| State / water behaviour | White crystalline solid; Water-insoluble at the test loading                                                                         |
| Acid-base pattern       | Neutral; ordinary dilute acid/base solubility tests negative                                                                         |
| Elements                | Nitrogen, sulphur and halogens negative in the teaching pattern                                                                      |
| Functional pattern      | Functional screens exclude the tested acid, phenol and carbonyl classes; supplied spectral evidence supports an aromatic hydrocarbon |
| Physical constant       | Melting region near 80°C                                                                                                             |
| Supported candidate     | Naphthalene                                                                                                                          |

**Worked Example**

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

- Record the candidate as Naphthalene only if your own results support the pattern. An anomalous physical constant should be investigated before final assignment.

**Result**

Complete the actual unknown record with its identification evidence and justified candidate.

**Precautions**

- Use instructor-assigned quantities and prepared test reagents in the designated laboratory; wear eye protection and use the required fume hood.
- Do not taste unknowns, smell containers directly, burn unknown halogenated material or heat sealed vessels.
- Use fresh separate portions for independent tests. A single colour reaction does not establish molecular identity.
- Do not prepare picrate derivatives in this student activity or use combustion as sole identification.

**Viva Questions and Answers****1. Can the title identify an actual unknown?**

No. The title describes this worked case; the actual material must be analysed.

**2. Why perform a confirmatory measurement?**

Different molecules can share solubility and functional-group reactions.

**3. What belongs in the result?**

The observed evidence, proposed identity and any unresolved limitation.

# Systematic Analysis of an Amino Ester

Experiment 12 - Practical Notes

Name: \_\_\_\_\_ Roll no.: \_\_\_\_\_ Date: \_\_\_\_\_

## Aim

To organise an illustrative unknown-identification record and show how the assigned candidate is supported.

## Principle

This is a worked teaching case, not an observation to copy into a laboratory record. An actual unknown is assigned by the instructor and may give different findings. A candidate is supported by consistent preliminary, elemental, functional and physical evidence.

## Requirements

- Instructor-assigned unknown and the corresponding known control; the method notes in this subject.
- Prepared test reagents, separate test portions and calibrated physical-constant apparatus.

## Procedure

### 1. Preliminary examination

Record the actual state and water behaviour at the prescribed quantity and temperature. Compare with the illustrative pattern below.

### 2. Solubility classification

Test fresh portions in the assigned acid/base reagents. Describe dissolution or effervescence before inferring a class.

### 3. Elements

Use the instructor-prepared extract or provided elemental report. Distinguish an element finding from a functional-group conclusion.

### 4. Functional confirmation

Instructor-led aromatic-primary-amine evidence plus the assigned ester reaction supports two functional groups

### 5. Physical confirmation

Use the dry-parent melting range and teacher-provided confirmatory spectrum or appropriate assigned derivative.

### 6. Conclude

Write the candidate only after reviewing all evidence; list a plausible alternative and the test that distinguishes it.

**Pharmaceutical Organic Chemistry****Observations and Record**

| Evidence                | Illustrative finding                                                                                           |
|-------------------------|----------------------------------------------------------------------------------------------------------------|
| State / water behaviour | White crystalline solid; Poor water solubility                                                                 |
| Acid-base pattern       | Can show increased solubility through protonation in the appropriate acid system                               |
| Elements                | Nitrogen positive; sulphur and halogens negative in the teaching pattern                                       |
| Functional pattern      | Instructor-led aromatic-primary-amine evidence plus the assigned ester reaction supports two functional groups |
| Physical constant       | Typical melting region about 88-90°C; compare with the actual batch standard                                   |
| Supported candidate     | Benzocaine                                                                                                     |

**Worked Example**

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

- Record the candidate as Benzocaine only if your own results support the pattern. An anomalous physical constant should be investigated before final assignment.

**Result**

Complete the actual unknown record with its identification evidence and justified candidate.

**Precautions**

- Use instructor-assigned quantities and prepared test reagents in the designated laboratory; wear eye protection and use the required fume hood.
- Do not taste unknowns, smell containers directly, burn unknown halogenated material or heat sealed vessels.
- Use fresh separate portions for independent tests. A single colour reaction does not establish molecular identity.
- A molecule can have more than one functional group; do not stop the analysis after detecting an amine.

**Viva Questions and Answers****1. Can the title identify an actual unknown?**

No. The title describes this worked case; the actual material must be analysed.

**2. Why perform a confirmatory measurement?**

Different molecules can share solubility and functional-group reactions.

**3. What belongs in the result?**

The observed evidence, proposed identity and any unresolved limitation.

# Building Ball-and-Stick Molecular Models

Experiment 13 - Practical Notes

Name: \_\_\_\_\_ Roll no.: \_\_\_\_\_ Date: \_\_\_\_\_

## Aim

To construct representative molecules and explain geometry, connectivity and selected isomerism.

## Principle

Ball-and-stick models represent atoms and bonds, while idealised local geometry is related to bonding. Single-bond rotation can produce conformers without changing connectivity; double bonds restrict rotation. Models are explanatory representations, not literal electron-density pictures.

## Requirements

- Molecular-model kit with colour key and bond connectors; instructor-assigned structures.
- Drawing sheet for structural formulas, geometry labels and conformer comparison.

## Procedure

### 1. Set the key

Record the atom-colour convention used by your kit rather than assuming every kit is identical.

### 2. Construct simple geometries

Build methane, ethene and ethyne. Compare tetrahedral, trigonal-planar and linear arrangements around the relevant carbons.

### 3. Build functional groups

Construct an alcohol, carboxylic acid, ester and amide. Show the atom connectivity and explain the group differences.

### 4. Aromatic model

Build benzene as a planar ring and discuss delocalisation; alternating model sticks are a representation, not fixed isolated double bonds.

### 5. Conformers and isomers

Rotate a single bond in ethane/butane and contrast this with cis/trans or E/Z possibilities for a suitable substituted alkene.

### 6. Draw the record

Translate each model into a clear structural formula and label the relevant geometry and functional group.

## Observations and Record

| Molecule / site | Idealised geometry  | Approximate bond angle |
|-----------------|---------------------|------------------------|
| Methane carbon  | Tetrahedral         | 109.5°                 |
| Ethene carbon   | Trigonal planar     | 120°                   |
| Ethyne carbon   | Linear              | 180°                   |
| Benzene ring    | Planar, delocalised | Near 120°              |

## Worked Example

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

- Ethene has  $C_2H_4$  with two trigonal-planar carbon centres. It does not have freely rotating C-C single-bond conformers.

## Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

## Precautions

- Follow valence requirements and do not add bonds merely to fill every connector hole.
- Distinguish a conformer from a constitutional isomer; model colours are conventions.

## Viva Questions and Answers

### 1. What changes in a conformer?

Spatial arrangement through permitted bond rotation without changing connectivity.

### 2. Why is ethyne linear?

The local carbon bonding geometry is approximately linear.

### 3. Does a benzene model prove fixed alternating bond lengths?

No. The ring is delocalised and the bond depiction is a teaching convention.

# Recrystallisation of Benzoic Acid

Experiment 14 - Practical Notes

Name: \_\_\_\_\_ Roll no.: \_\_\_\_\_ Date: \_\_\_\_\_

## Aim

To purify benzoic acid by recrystallisation and calculate mass recovery.

## Principle

A useful crystallisation solvent dissolves much more of the compound when hot than when cold. Hot insoluble impurities can be removed before cooling; soluble impurities should largely remain in the mother liquor. Recovery and purity are separate outcomes.

## Requirements

- Assigned impure benzoic acid, water and the instructor's approved microscale method.
- Flask, controlled heater/bath, appropriate hot-filtration apparatus, vacuum filtration, drying setup and melting-point instrument.

## Procedure

### 1. Weigh

Record the mass of the impure solid and prepare clean labelled vessels.

### 2. Dissolve

Add the selected solvent in small increments while heating under the assigned method until the solid just dissolves. Avoid vigorous bumping and retain the least hot water needed for dissolution.

### 3. Clarify

If hot insoluble matter is present, use the teacher-approved warmed filtration setup. Activated carbon is used only when justified and in a small assigned quantity.

### 4. Crystallise

Allow the clear solution to cool slowly undisturbed, then use the prescribed final cooling step. Record crystal formation instead of assuming full recovery.

### 5. Collect

Filter the crystals, wash with a small amount of cold appropriate solvent and dry to the specified condition.

### 6. Assess

Weigh the dry crystals, calculate recovery and measure melting range. Record whether the range narrows compared with the starting material; excessive solvent can reduce recovery.

**Pharmaceutical Organic Chemistry**

---

**Observations and Record**

| Record                          | Entry                            |
|---------------------------------|----------------------------------|
| Starting mass / solvent volume  | _____                            |
| Dry recovered mass / recovery   | _____                            |
| Before and after melting ranges | _____                            |
| Expected comparison             | Typical parent region: 121-123°C |

**Worked Example**

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

- Starting mass 1.00 g and dry recovered mass 0.72 g gives recovery = 72.0%. This is not a reaction yield or a direct purity assay.

**Result**

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

**Precautions**

- Use instructor-assigned quantities and prepared test reagents in the designated laboratory; wear eye protection and use the required fume hood.
- Do not taste unknowns, smell containers directly, burn unknown halogenated material or heat sealed vessels.
- Use fresh separate portions for independent tests. A single colour reaction does not establish molecular identity.
- Keep a vapour escape path and use the approved heating source; never heat a sealed crystallisation vessel.

**Viva Questions and Answers****1. Why minimise hot solvent?**

Excess solvent retains more compound after cooling and can reduce recovery.

**2. Why wash with cold solvent?**

It removes adhering mother liquor while limiting dissolution of the crystals.

**3. Does high recovery prove purity?**

No. Impurities can also increase the recovered mass.

# Recrystallisation of Acetanilide

Experiment 15 - Practical Notes

Name: \_\_\_\_\_ Roll no.: \_\_\_\_\_ Date: \_\_\_\_\_

## Aim

To purify acetanilide by recrystallisation and calculate mass recovery.

## Principle

A useful crystallisation solvent dissolves much more of the compound when hot than when cold. Hot insoluble impurities can be removed before cooling; soluble impurities should largely remain in the mother liquor. Recovery and purity are separate outcomes.

## Requirements

- Assigned impure acetanilide, water and the instructor's approved microscale method.
- Flask, controlled heater/bath, appropriate hot-filtration apparatus, vacuum filtration, drying setup and melting-point instrument.

## Procedure

### 1. Weigh

Record the mass of the impure solid and prepare clean labelled vessels.

### 2. Dissolve

Add the selected solvent in small increments while heating under the assigned method until the solid just dissolves. Its low cold-water solubility makes a hot-water/cooling comparison useful; warm filtration apparatus helps avoid premature crystallisation.

### 3. Clarify

If hot insoluble matter is present, use the teacher-approved warmed filtration setup. Activated carbon is used only when justified and in a small assigned quantity.

### 4. Crystallise

Allow the clear solution to cool slowly undisturbed, then use the prescribed final cooling step. Record crystal formation instead of assuming full recovery.

### 5. Collect

Filter the crystals, wash with a small amount of cold appropriate solvent and dry to the specified condition.

### 6. Assess

Weigh the dry crystals, calculate recovery and measure melting range. Record whether the range narrows compared with the starting material; excessive solvent can reduce recovery.

## Observations and Record

| Record                          | Entry                                                              |
|---------------------------------|--------------------------------------------------------------------|
| Starting mass / solvent volume  | _____                                                              |
| Dry recovered mass / recovery   | _____                                                              |
| Before and after melting ranges | _____                                                              |
| Expected comparison             | Typical parent region: 113-116°C, depending on method and standard |

## Worked Example

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

- Starting mass 1.00 g and dry recovered mass 0.72 g gives recovery = 72.0%. This is not a reaction yield or a direct purity assay.

## Result

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

## Precautions

- Use instructor-assigned quantities and prepared test reagents in the designated laboratory; wear eye protection and use the required fume hood.
- Do not taste unknowns, smell containers directly, burn unknown halogenated material or heat sealed vessels.
- Use fresh separate portions for independent tests. A single colour reaction does not establish molecular identity.
- Keep a vapour escape path and use the approved heating source; never heat a sealed crystallisation vessel.

## Viva Questions and Answers

### 1. Why minimise hot solvent?

Excess solvent retains more compound after cooling and can reduce recovery.

### 2. Why wash with cold solvent?

It removes adhering mother liquor while limiting dissolution of the crystals.

### 3. Does high recovery prove purity?

No. Impurities can also increase the recovered mass.

# Recrystallisation of Naphthalene

Experiment 16 - Practical Notes

Name: \_\_\_\_\_ Roll no.: \_\_\_\_\_ Date: \_\_\_\_\_

## Aim

To purify naphthalene by recrystallisation and calculate mass recovery.

## Principle

A useful crystallisation solvent dissolves much more of the compound when hot than when cold. Hot insoluble impurities can be removed before cooling; soluble impurities should largely remain in the mother liquor. Recovery and purity are separate outcomes.

## Requirements

- Assigned impure naphthalene, the assigned ethanol-water mixed-solvent system and the instructor's approved microscale method.
- Flask, controlled heater/bath, appropriate hot-filtration apparatus, vacuum filtration, drying setup and melting-point instrument.

## Procedure

### 1. Weigh

Record the mass of the impure solid and prepare clean labelled vessels.

### 2. Dissolve

Add the selected solvent in small increments while heating under the assigned method until the solid just dissolves. The instructor controls the mixed-solvent sequence; use water as the poorer solvent only as directed and avoid an oiling-out condition.

### 3. Clarify

If hot insoluble matter is present, use the teacher-approved warmed filtration setup. Activated carbon is used only when justified and in a small assigned quantity.

### 4. Crystallise

Allow the clear solution to cool slowly undisturbed, then use the prescribed final cooling step. Record crystal formation instead of assuming full recovery.

### 5. Collect

Filter the crystals, wash with a small amount of cold appropriate solvent and dry to the specified condition.

### 6. Assess

Weigh the dry crystals, calculate recovery and measure melting range. Record whether the range narrows compared with the starting material; excessive solvent can reduce recovery.

**Pharmaceutical Organic Chemistry**

---

**Observations and Record**

| Record                          | Entry                            |
|---------------------------------|----------------------------------|
| Starting mass / solvent volume  | _____                            |
| Dry recovered mass / recovery   | _____                            |
| Before and after melting ranges | _____                            |
| Expected comparison             | Typical parent region: near 80°C |

**Worked Example**

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

- Starting mass 1.00 g and dry recovered mass 0.72 g gives recovery = 72.0%. This is not a reaction yield or a direct purity assay.

**Result**

Record the observed outcome and calculated value, with units. State whether the stated laboratory specification was met; do not copy the worked example as your result.

**Precautions**

- Use instructor-assigned quantities and prepared test reagents in the designated laboratory; wear eye protection and use the required fume hood.
- Do not taste unknowns, smell containers directly, burn unknown halogenated material or heat sealed vessels.
- Use fresh separate portions for independent tests. A single colour reaction does not establish molecular identity.
- Keep a vapour escape path and use the approved heating source; never heat a sealed crystallisation vessel.

**Viva Questions and Answers****1. Why minimise hot solvent?**

Excess solvent retains more compound after cooling and can reduce recovery.

**2. Why wash with cold solvent?**

It removes adhering mother liquor while limiting dissolution of the crystals.

**3. Does high recovery prove purity?**

No. Impurities can also increase the recovered mass.

# Continue Learning with FirstHope

## Complete Notes on the FirstHope App

**Complete subject and semesters are available on FirstHope app only.**

- Get complete subject and semester notes in the FirstHope app.
- Available for B.Pharm, D.Pharm, M.Pharm and Pharm.D students.

**[Click here to get the FirstHope app](#)**

## Join the FirstHope Telegram Channel

Join FirstHope Notes and keep up with channel updates.

**[Click here to join Telegram](#)**

## Instagram

@firsthope.official

**[Click here to follow Instagram](#)**

## YouTube Channel

FirstHope-Pharmacy / Live streams

**[Click here to watch the YouTube channel](#)**

## WhatsApp Channel

Follow FirstHope on WhatsApp.

**[Click here to join the WhatsApp channel](#)**

## FirstHope Website

firsthopenotes.com

**[Click here to visit the website](#)**