LAB REPORT INSTRUCTIONS

Identifying an Unknown Compound by Infrared Spectroscopy (IR)
Carefully read all instructions and complete Section I on page 3 before you come to the lab.

Background

- The primary use of IR spectroscopy is functional group identification. Organic compounds absorb energy in the infrared region (IR) of the electromagnetic spectrum.
- Bonds are not rigid links—rather, one can compare them to tiny “springs” with varying degrees of stiffness that connect the atoms together.
- These vibrations include: stretching, bending, twisting, etc.

- Infrared energy can cause the atoms of organic compounds to vibrate faster and with increased amplitude about the covalent bonds that connect them.
- The most important factors determining where a chemical bond will absorb in the IR region are:
  - The bond order (single, double, or triple bond)—i.e., the stiffness of the spring
  - The types of atoms joined by the bond (i.e., the mass (m₁, m₂)
- The absorption frequencies for different organic functional group in IR spectrum are shown below.

\[ \text{Wave number, cm}^{-1} \]

\[ \text{Wavelength, microns} \]
IR Interpretation:

**Step 1: Unsaturation / Elements of Unsaturation / Degrees of Unsaturation / Index of Hydrogen Deficiency:**

Use the molecular formula to calculate the number of unsaturations (rings or multiple bonds). It limits the number of possible structures when you look at the IR spectrum. The degree of unsaturation indicates whether or not the compound has one or more double bonds or rings, or even a triple bond. There are different ways to calculate it.

*Note the following relationship between carbon and hydrogen: alkanes - \( C_nH_{2n+2} \); alkenes and cyclohexane - \( C_nH_{2n} \); alkynes - \( C_nH_{2n-2} \). Try these formulae by analyzing ethane, ethene, and ethyne.*

Always compare to the formula of alkanes, \( C_nH_{2n+2} \).

Note the following rules when heteroatoms (atoms other than carbon and hydrogen) are present:

- **Organohalogenes**, containing C, H, X where X = F, Cl, Br, I; organooxygen compounds, containing C, H, O; and organonitrogen compounds, containing C, H, N. Of course there are molecules that have all of the above, *i.e.* C, H, O, X, N.

Take the formula that you are given and:

- **Add** the number of halogens to the number of hydrogens
- **Ignore** the number of oxygens
- **Subtract** the number of nitrogens from the number of hydrogens

For example: \( C_5H_9\text{N} \) is equivalent to “\( C_5H_8 \)”. Compare the latter with a fully saturated molecule. According to \( C_nH_{2n+2} \) an alkane with 5 carbons should have 12 hydrogens. So, half of the hydrogen difference gives you the element(s) / degree(s) of unsaturation: \( (12-8)/2 = 2 \). Thus, \( C_3H_9\text{N} \) has two degrees of unsaturation and could contain one of the following: a) a triple bond; b) two double bonds or c) one double bond and a ring.

Or take the formula and use the equation below:

- Number of Unsaturations = \( \{2(#C) + 2 - #H - #\text{Hal} + #N\}/2 \)

For example: \( C_5\text{H}_9\text{N} \) would yield: \( \{2x5 + 2 - 9 - 0 + 1\}/2 = 2 \)

**Step 2: Analyzing the IR Spectrum:**

Look at the IR spectrum, especially in the region greater than 1500 cm\(^{-1}\) (outside the “Fingerprint region” which is below 1500 cm\(^{-1}\)). Look for likely functional groups (O-H, N-H, C-H, C≡N, C=O, C=C, C-O), working in conjunction with the calculated number of unsaturations and the molecular formula. For instance, if there is one unsaturation and an oxygen, it could be a carbonyl; if there is no unsaturation and an oxygen, it will be either an ether or an alcohol; if there are 4 degrees of unsaturation, there might be an aromatic ring, etc. Also note that primary, secondary and tertiary amines can be distinguished by the number of spikes between ca 3200-3500 (RNH\(_2\) – two spikes, R\(_2\)NH – one spike, R\(_3\)N – no spike for any nitrogen with no hydrogen attached). Avoid the temptation to assign every peak; only focus on the most important regions and key stretches.

**MOST IMPORTANT THING TO REMEMBER…..**

When analyzing an IR spectrum **avoid the temptation to try to assign every peak**... Some examples…

![IR Spectrum Diagrams](image-url)
Experimental Notes

You must read and be familiar with the entire procedure **before** coming to lab. Do not expose IR salts and cells to water or even moisture from your hands. Closely follow the TA’s instructions for handling IR samples and the FT-IR spectrophotometer.

You will learn several new techniques:

1) How to prepare an IR sample of a **liquid** using KBr or NaCl plates.
2) How to prepare an IR sample of a **solid** using a KBr hand press.
3) How to analyze IR spectra.

Lab Report Notes

*(written in your lab notebook, 50 pts total)*

*You may work with a lab partner for the in-lab portion. The lab report is due at the beginning of the next lab session and it needs to reflect the student’s own work, no group work for lab report entries.*

*Print clearly in your lab notebook so that the carbon copies are legible. Label everything with your name, the date, and experiment number when you hand in the duplicate pages.*

**Section 1  Introduction** *(prepare this section **before** coming to the lab, **10 pts)*

1. (1 pt) Date and Title of Experiment
2. (1 pt) Objective (2 sentences)
3. Possible unknowns for this lab:
   a) Copy the structures into your lab notebook.
   b) Determine the degree of unsaturation for each.
   c) Assign the molecular formula for each.
   d) Identify major functional groups and review the wavenumber list.
Benzyl alcohol  Cyclohexane  Cyclohexene  Butyraldehyde  Isopentyl acetate

Benzonitrile  Cyclopentanol  1-Hexene  1-Heptyne

4. (5 pts) List the five possible properties of your unknown.

5. (1 pt) Benzoic acid and KBr are ____________________.

6. (1 pt) Why is it essential that you not handle KBr or NaCl plates with your bare hands?

7. (1 pt) Describe the procedure for preparing a liquid sample for infrared examination.

- Your TA will demonstrate how to prepare an IR sample of a solid and a liquid compound (record instructions into your lab notebook).
- Your TA will assign whether you will work on Part A or Part B first.
- Your TA will assign your unknown.

Section II  Observations and calculations (data recorded during the lab, 35 pts)

Part A: Preparation and measurement of an IR sample (data recorded during the lab, 20 pts)

Use a two-column outline
Left-brief, bulleted outline of procedure;
Right-briefly state your observations and comments for each step of the procedure

Obtain a sample of the unknown that your TA assigned.

8. (1 pt) Write down the unknown ID and molecular formula into your lab notebook.

9. (1 pt) Observe the physical properties of the unknown compound (liquid, solid, color, etc).

Proceed to the instrument room

10. Prepare an IR sample.

11. Obtain an IR spectrum.

12. Clean up. Do NOT rinse the IR cells with water or acetone. Use the appropriate solvent.

Return to your lab bench

13. (1 pt) Label the spectrum with your name, unknown ID, formula.

14. (2 pt) Calculate the degree of unsaturation.

15. (2 pt) Based on your findings under #14, write down one or more possible functional groups.

16. (3 pts) What is the solubility of your unknown in a) water, b) ethanol, and c) tert-butylmethyl ether.

17. (2 pts) What do you conclude from your observations under #16 using the concept of “like dissolves like”?

18. (3 pts) Label all of the pertinent functional groups that you observe in the IR of your unknown. Prepare a chart in your laboratory notebook indicating the absorption frequency and structural assignment (e.g. 3350 cm⁻¹, O-H stretch).

19. (5 pts) Identify the structure of your unknown. Explain your conclusion.
Part B: Spectral Interpretation of benzoic acid and \( p \)-anisaldehyde (data recorded during the lab, 15 pts)

Use a two-column outline
Left: brief, bulleted outline of procedure;
Right: briefly state your observations and comments for each step of the procedure

20. Draw the structures of benzoic acid and \( p \)-anisaldehyde.
21. (2 pts) What are the physical characteristics of benzoic acid, \( p \)-anisaldehyde and your unknown (solid, liquid, colored, etc., and include safety data)?
22. (6 pts) What is the solubility of benzoic acid and \( p \)-anisaldehyde in a) water, b) ethanol, and c) \( \text{tert} \)-butylmethyl based on the concept of “like dissolves like.”?
23. (3 pts) Clearly label the IR spectrum of benzoic acid and assign the following peaks (absorption frequencies are approximate): broad 3300-2500 cm\(^{-1}\), 3020 cm\(^{-1}\), 1682 cm\(^{-1}\).
24. (4 pts) Clearly label the spectrum of anisaldehyde and assign the following peaks (absorption frequencies are approximate): 3074 & 3008 cm\(^{-1}\), 2965 cm\(^{-1}\), 2834 & 2736 cm\(^{-1}\), and 1695 cm\(^{-1}\).

Section III Results and Discussion (analysis after the experiment is completed, 4 pts)

25. (1 pt) What other methods could you use to help confirm the identity of your unknown?
26. (2 pt) Although IR spectra of solids are most commonly run as KBr pellets, they are sometimes run as solutions. Even though most organic compounds are more soluble in acetone than they are in tetrachloromethane (CCl\(_4\)), acetone is almost never used as a solvent in running IR spectra of solids. However, CCl\(_4\) is very acceptable provided that the solvent will dissolve in it. Briefly explain why CCl\(_4\) is preferred over acetone.
27. (1 pt) Explain why CH\(_2\)Cl\(_2\) is preferred over CCl\(_4\) (think safety)?

Section IV Conclusions (1 pts)

List two new things you learned in doing this experiment or something that you now understand better than before you did the lab. Explain.

_____________________________
Sign & date your lab report

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