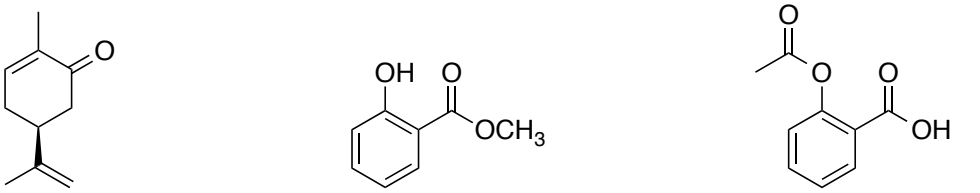
## Experiment 4 – IR Exercise

Reading Assignment
Mohrig Chapter 21 and watch IR videos online

In this experiment, students will study the infrared (IR) spectra of compounds with different functional groups. The purpose of the experiment is to become familiar with the IR spectrometer, the preparation and handling of IR samples, and the interpretation of IR spectra. CHEM 8M students use the IR nearly every lab. You will obtain the IR spectra of aspirin, carvone, and methyl salicylate (wintergreen oil). Carvone and methyl salicylate are liquids and their IRs are obtained directly from a very small drop of pure (aka "neat") material. Aspirin is a solid and must be diluted before the IR is obtained.




**Carvone** | **Methyl Salicylate (Wintergreen Oil)** | **Acetylsalicylic Acid (Aspirin)**

**Figure 1.** Structures of compounds to be analyzed by IR.

IR spectroscopy is similar to spectrophotometry, a technique that is used in the general chemistry labs to determine concentration of samples based on absorbance of visible light. In spectrophotometry, a colored sample absorbs light in the visible range of the electromagnetic spectrum. IR spectroscopy explores a different range of frequencies in order to determine the types of bonds and functional groups present but does not require a visible color in the compound. Absorption of IR radiation results in the stretching and bending of bonds in ways characteristic to the functional group. Before beginning to prepare the notebook for this lab, ***read the sections on IR in the Mohrig text and attend the IR lecture! You may bring this handout to lab to compare with literature IR, but you still need to prepare your lab notebook or you will not be permitted to enter the lab (zero points on the report).***

**Notebook Preparation**

- *Purpose:* Describe the purpose of the experiment in one sentence. Draw the structures of carvone, methyl salicylate, and aspirin at the top of the page. Circle and name each of the functional groups present in each molecule.
- *Reagent Table:* List the properties for each compound (MW, bp/mp, density, safety notes).
- *Written Procedure*, as described below. **Include 3 IR tables**, one for each compound, where at least the first 3 columns are completed. **You will not be permitted in the lab without these tables!**
- *Safety & Cleanup:* Copy table into your notebook.

**PROCEDURE** – students work in pairs

*Logistics:* There is only one IR and two IR kits per lab so students will take turns. Please be patient and use your down time constructively (no cell phones used in the lab). Some lab rooms may need to share one instrument so pay attention to instructions on creating a queue for using the IR. While you're waiting, compare your IR tables with your lab mates, read the text, prepare for the next experiment, etc. If there aren't too many people in the instrument room, you can observe the TA demo with another group and help in sample preparation or cleanup. Don't worry, there is plenty of time for each group to run their samples and analyze the data.

*Overview:* Your TA will demonstrate how to operate the spectrometer. Under TA supervision, obtain the IR spectra of carvone, methyl salicylate, and aspirin. You will use salt plates (pure NaCl) to support your sample. Sodium chloride does not absorb IR radiation making it an ideal material to hold samples. The NaCl plates are very fragile and break easily. Handle them with care and never wash them with water (they will dissolve!). Instead, use small amounts of the acetone saturated with NaCl provided in the IR kit.

*IR of liquid samples, carvone and methyl salicylate:* Touch the liquid with the tip of a pipet to pick up a small drop of liquid then touch the center of the salt plate with the tip of the pipet, using your thumb to apply pressure. This small amount of liquid should be enough to obtain a good IR. Cover the plate with the other half and spread the liquid by rotating the plates. Place the plates inside the plate holder, being careful not to break the plates, as demonstrated by your TA and obtain the IR spectrum. A "nice looking" IR will contain bands ending in sharp peaks rather than being flat at the bottom. Flat signals are a result of too much sample being used.

*IR of a solid sample:* Place a microspatula-full of the solid in a mortar and add just one drop of Nujol. Grind the mixture with a pestle for about a minute to get a dense paste (aka "Nujol mull"). Grinding the solid very well is necessary since big solid particles will scatter IR light and lead to curved baselines and distorted spectra. Scoop some of the mull with the rubber policeman provided in the IR kit and spread it on one of the salt plates. Cover with the other plate and rotate them to further spread the mull. Obtain the IR the same way you did for the liquid sample. Keep in mind that Nujol absorbs IR radiation as well. Take a look at the IR of Nujol for reference and to avoid confusing Nujol peaks for sample peaks.

*Analysis:* First, do a quick comparison your "observed" IR's with the "literature" IRs posted online. They should look similar to each other, though they may not be identical. If they look strikingly different, consult your TA to diagnose the problem (too much sample or Nujol, too little sample, etc.). You can refer to the end of the IR chapter in the Mohrig text for common IR issues. Run the IR again if necessary, time permitting. If the IR looks good, complete the detailed analysis of each compound and its corresponding spectra by completing a table formatted as follows. *Before coming to lab, draw three tables like the one below into your notebook for each of the three compounds you will analyze. Leave extra space in the cells in case you need to change your answer – use a single-line strike-through and draw your corrected answer above.* Refer to the "Interpreting IR Spectra" section in the Mohrig text for guidance.

**Table x.** IR Analysis of (Compound Name, *redraw structure if it's not on that page already)

| Functional Group | Bond Assignment (C=O, N-H, etc.) from IR Table | Expected Wavenumber from IR Table | Literature Wavenumber (IR online) | Observed Wavenumber (your IR) |
|---|---|---|---|---|
| | | | | |
| | | | | |
| | | | | |

Begin filling in these tables before lab. Use the IR Table handed out in lecture (also posted online) to determine the expected peaks. **With the exception of the "Observed Wavenumber" column, everything else can be filled in based on theoretical values and the sample IRs provided online.**

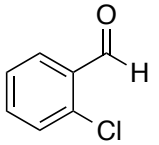
Since this is a new technique, you will not be graded on the accuracy of what you walk into lab with, but *you must have the tables in your notebook or you will not be permitted in the lab.* Use the format above along with the example IR table on the following page and give it a solid college try! Review your work with your lab partner. If your tables are messy or difficult to read, it is recommended you re-draw the tables to submit with your report.

**You are strongly encouraged to attend your TA's office hours (or Dr. B's) before this lab to get ahead of the game so plan accordingly! Please review your lecture notes and the background reading carefully before asking for help. When you do your part, you are helping us help you!**

*Copy these safety and cleanup notes into your notebook…*

| **Safety First!** | **Clean- up** |
|---|---|
| - Wear gloves when preparing the sample using mortar & pestle. Gloves should be changed often and removed immediately after completion of the chemical operation. | - Clean the salt plates, the mortar and pestle, and the rubber policeman with a little acetone (sat. w NaCl) and tissue paper. Wear gloves when cleaning and remove when you're done. |
| - Methyl salicylate is toxic. | - Dispose of tissue paper in the trash and **pipets in solid-waste**. |
| - Carvone is an irritant | |

*Example (for your reference, no need to include in notebook)…*




***o*-Chlorobenzaldehyde**

Functional Groups: Aromatic Ring, Aldehyde, Aryl Chloride

**Table 1.** IR Analysis of *o*-chlorobenzaldehyde (structure above)

| **Functional Group** | **Bond Assignment (C=O, N-H, etc.) from IR Table** | **Expected Wavenumber, $cm^{-1}$ from IR Table** | **Literature** Wavenumber, $cm^{-1}$ (from the IR provided)** | **Observed* Wavenumber (your IR)** |
|---|---|---|---|---|
| Aromatic ring o-disub | C-H stretch | 3100 – 3000 | 3070 | |
| | C=C | 1625 – 1440 | ~1600† | |
| | C-H bend | 900 – 680 (770 – 735)ø | 760 | |
| Aldehyde (conj.) | C-H stretch | 2900 – 2800 & 2800 –2700, doublet | 2868 & 2753 | |
| | C=O | 1715 – 1680 | 1695 | |
| Aryl Chloride | C-Cl | < 600 – 840 | ~800† | |

* You will not take an IR of *o*-chlorobenzaldehyde so there is nothing to fill in for these columns. This is just an example. For the compounds that you will analyze, this column will be blank until you obtain your own IR in lab.

** Refer to the literature IR provided at the end of this handout.

ø Specific out-of-plane bending vibrations (Table 2 in IR reference sheet)

† Values approximated from the x-axis of the IR spectrum.

**Introduction: Pre-lab Questions** – typed, printed, and turned in at the beginning of lab

1. What happens when IR radiation is absorbed by an organic sample? How is the frequency of the radiation used to determine the functional groups in the molecule?

2. In IR spectroscopy, we normally talk about "frequencies" when in reality we are referring to wavenumbers. What is the mathematical relationship between frequency and wavenumber? Between wavenumber and wavelength? What are the units most commonly used for frequency, wavelength, and wavenumber?

3. What is the range for the IR fingerprint region? Why are the bands in this region of limited use in structure elucidation?

4. What is Nujol (look it up online)? Where (what wavenumbers) does it absorb IR radiation (see attached IR)?

5. Is the ketone in carvone classified as aliphatic, conjugated, or aromatic?

**Results: In-lab Questions**

1. (*30 points*) Complete the IR tables as described in the procedure. Remember that it's not necessary to assign every peak on the IR spectrum and that not all expected absorbances may be observed. Give each table a number and descriptive title. Be sure to discuss your results with your lab partner and TA before leaving lab.

2. (*5 points*) List one or two of the most distinctive functional groups for each compound and corresponding absorbance(s). In other words, what is the easiest way to tell these IRs apart?

** Attach the IR spectra to the back of the report.

References

Mohrig, J. R.; Hammond, C. N.; Schatz, P. F. "Infrared Spectroscopy" in *Techniques in Organic Chemistry*. Freeman: New York, **2006**.

Palleros, D. R. "Infrared Spectroscopy" in *Experimental Organic Chemistry*. Wiley: New York, **2000**.

**Exp 4 – IR Exercise** Name _____
*Due date in the syllabus*

Section Day _____ Time _____ TA Name _____

CHEM 8L GRADING RUBRIC - Use as cover page for report

| SECTION | INSTRUCTOR COMMENTS | POINTS ASSIGNED |
|---|---|---|
| **IN-LAB QUIZ** | | **/ 5** |
| **LAB REPORT** | | |
| **INTRODUCTION**<br>Each pre-lab question is numbered and addressed using complete sentences. Structures and calculations are hand-written, where appropriate. | | / **25** |
| **RESULTS**<br>The main results are stated, as outlined in the in-lab questions, using complete sentences and/or tables. | | / **35** |
| **NOTEBOOK PAGES**<br>Proper format: chemical structures, chemical info table, procedure, waste and clean-up procedure. | | / **10** |
| **CALCULATIONS & IR WORKSHEET**<br>Individual responses complete and correct. Shown to the TA for credit before the end of lab – not turned in. | | / **10** |
| **LAB TECHNIQUE & CLEAN UP**<br>Instrument room and IR kits left clean, proper technique, instructions followed, checked in with TA before leaving.<br>Proper order & format.<br>IR spectra attached. | | / **15** |
| | **LAB REPORT TOTAL** | **/ 100** |