

MORAVIAN COLLEGE CHEMISTRY DEPARTMENT

Chemistry 211L

**Organic Chemistry
Semester 1**

LABORATORY MANUAL

Fall Semester 2013

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PART I: Introduction:*CHEM 211L Fall 2013 Laboratory Schedule*

Week 1 Laboratory Discussion (Monday, August 26)	
Assignment:	What is POGIL? Activity Sheet
Topic:	What is POGIL and why are we using it?
Laboratory Periods (Tu-F, August 27-30)	
Activity:	Check-in, Safety Orientation and the Interpretation of Data Activity
Assignment:	Lab Manual: Introduction & Rules and Regulations. (pp. 3-12).
Week 2 Laboratory Discussion (Friday, August 30)	
Assignment:	Experiment 1: How is the Boiling Behavior of a Liquid Molecule Related to Its Molecular Structure and Purity? <i>Parts A & B</i> (pp. 20-22) Pre-lab by 10:00 PM Thurs. Aug. 29 (Course Website)
Topic:	Part A: <i>How does overall molecular structure determine the boiling point of a compound?</i> Part B: <i>How can we determine the purity of a liquid sample?</i>
Laboratory Periods (Tu-Th, September 3-6)	
Activity:	Experiment 1 Part A: <i>How does overall molecular structure determine the boiling point of a compound?</i>
Week 3 Laboratory Discussion (Friday, September 6)	
Assignment:	Experiment 1: How is the Boiling Behavior of a Liquid Molecule Related to Its Molecular Structure and Purity? <i>Part B</i> (pp. 23-24) Pre-lab by 10:00 PM Thurs. Sept. 5 (Course Website)
Topic:	<i>How can we determine the purity of a liquid sample?</i>
Laboratory Periods (Tu-F, September 10-13)	
Activities:	Experiment 1 Part A: <i>How does overall molecular structure determine the boiling point of a compound?</i> Data analysis Experiment 1 Part B: <i>How can we determine the purity of a liquid sample?</i> Data collection

Week 4 Laboratory Discussion (Monday, September 16) Assignment: Experiment 2: How Does the pH of Aqueous Solutions Affect the Solubilities of Organic Compounds? <i>Part A:</i> (p. 25-26) Pre-lab by 10:00 PM Sun. Sept. 15 (Course Website) Topic: <i>What can solubility in acidic and basic aqueous solutions tell us about structures of organic molecules?</i>	
Laboratory Periods (Tu-F, September 17-20) Activity: Experiment 2: How Does the pH of Aqueous Solutions Affect the Solubilities of Organic Compounds? <i>Part A:</i>	
Week 5 Laboratory Discussion (Monday, September 23) Assignment: Experiment 2: How Does the pH of Aqueous Solutions Affect the Solubilities of Organic Compounds? <i>Part B:</i> (p. 27-28) Pre-lab by 10:00 PM Mon. Sept. 22 (Course Website) Topic: <i>How can we use pH effects on solubility to separate mixtures of compounds?</i>	
Laboratory Periods (Tu-F, September 24-27) Activity: Experiment 2: How Does the pH of Aqueous Solutions Affect the Solubilities of Organic Compounds? <i>Part B:</i> Due: Report on Experiment 1 due at the end of the lab period.	
Week 6 Laboratory Discussion (Monday, September 30) Assignment: Experiment 3: How is the Melting Behavior of a Mixture of Solids Related to those of the Separate Pure Solids used to create the mixture? (pp. 29-30) Pre-lab by 10:00 PM Sun. Sept. 29 (Course Website) Topic: <i>How is the melting behavior of a mixture of solids related to the melting behavior of the two pure solids used for the mixture?</i>	
Laboratory Periods (Tu-F, October 1-4) Activity: Experiment 3: How is the Melting Behavior of a Mixture of Solids Related to those of the Separate Pure Solids used to create the mixture? Due:	

Week 7 Laboratory Discussion (Monday, October 7) Assignment: Experiment 4: How Can We Judge the Purity of an Unknown Solid Sample? (pp. 31-32) Pre-lab by 10:00 PM Sun. Oct. 6 (Course Website) Topic: How Can We Judge the Purity of an Unknown Solid Sample?	
Laboratory Periods (Tu-F, October 8-11) Activity: Experiment 4: How Can We Judge the Purity of an Unknown Solid Sample? Due: Report on Experiment 2 due at the end of the lab period.	
Week 8 Laboratory Discussion (Friday, October 11) Assignment: Experiment 5: How Can a Crude Solid Substance Be Purified? (pp. 33-34) Pre-lab-1 by 10:00 PM Thurs. Oct. 10 (Course Website) Topic: How Can a Crude Solid Substance Be Purified?	
Laboratory Periods (W, Th, F & Tu, October 16, 17, 18 & 22) Activity: Experiment 5: How Can a Crude Solid Substance Be Purified? Due: Lab Report on Experiment 3 due at the end of the lab period.	
Week 9 Laboratory Discussion (Tuesday, October 22) Assignment: Experiment 6: How Can We Tell if Two Solid Organic Compounds Are the Same or Different? (pp. 35-36) Pre-lab by 10:00 PM Mon. Oct. 21 (Course Website) Topic: <i>What measurements can allow us to distinguish one compound from another?</i>	
Laboratory Periods (W, Th, F & Tu, October 23, 24, 25 & 29) Activity: Experiment 6: How Can We Tell if Two Solid Organic Compounds Are the Same or Different? Due: Lab report for Experiment 4 due at the end of the lab period	

Week 10 Laboratory Discussion (Tuesday, October 29) Assignment: Experiment 7: How Can We Determine the Structure of an Organic Compound? <i>Part A</i>: (pp. 37-38) Pre-lab by 10:00 PM Mon. Oct. 28 (Course Website) Topic: <i>How do organic compounds interact with high energy electrons and what structural information can we obtain from the results?</i>	
Laboratory Periods (W, Th, F & Tu, Oct. 30, 31, Nov. 1 & 5) Activity: Experiment 7: How Can We Determine the Structure of an Organic Compound? <i>Part A</i>: Due: Lab report for Experiment 5 due at the end of the lab period	
Week 11 Laboratory Discussion (Tuesday, November 5) Assignment: Experiment 7: How Can We Determine the Structure of an Organic Compound? <i>Part B: Week 1</i>: (p. 39-40) Pre-lab-1 by 10:00 PM Mon. Nov. 4 (Course Website) Topic: <i>How do organic compounds interact with light and magnetic fields and what structural information can we obtain from the results?</i>	
Laboratory Periods (W, Th, F & Tu, November 6, 7, 8 & 12) Activity: Experiment 7: How Can We Determine the Structure of an Organic Compound? <i>Part B: Week 1</i>: Due: Report on Experiment 6 due at the end of the lab period.	
Week 12 Laboratory Discussion (Tuesday, November 12) Assignment: Experiment 7: How Can We Determine the Structure of an Organic Compound? <i>Part B: Week 2</i>: (p. 41-42) Pre-lab-2 by 10:00 PM Mon. Nov. 11 (Course Website) Topic: <i>What structural information can be obtained from NMR spectra without chemical shift assignments?</i>	
Laboratory Periods (W, Th, F & Tu, November 13, 14, 15 & 19) Activity: Experiment 7: How Can We Determine the Structure of an Organic Compound? <i>Part B: Week 2</i>:	

Week 13	
Laboratory Discussion	
No Electronic Pre-lab	
Regular Class Group Activity Work	
Laboratory Periods (Tu-F, Nov. 19-22)	
Activity:	Experiment 7: How Can We Determine the Structure of an Organic Compound? <i>Part B: Week 3</i>
Week 14	
Laboratory Discussion	
(Monday, December 3)	
No Electronic Pre-lab	
Regular Class Group Activity Work	
Laboratory Periods (Tu-Th, Dec. 4-6)	
Activity:	Group Presentations of Reports on Experiment 7. and Check Out

Laboratory Philosophy (See also Padías pp. vi & 1-4)

Your organic chemistry laboratories, CHEM 211L and CHEM 212L are an integral part of the course. As with the class periods, the laboratory is also a “discovery” based experience. It will sometimes introduce you to new concepts and other time will expand on some of the concepts first developed in class. The laboratory will also introduce you to some of the research techniques that produce the data we discuss in class. You should consider the class and laboratory as two slightly different but equally important views of the area of science we call organic chemistry.

The primary focus of the laboratory is to give you experience with solving problems using data you collect in the laboratory. In each experiment, you will start with a question, develop an experimental plan to answer it, collect data and analyze the data to develop an answer (claim) and support your claim with a warrant.

Your work in CHEM 211L will often involve collaboration with members of a “research group”. Although you will usually have individual tasks to perform and specific data to collect, your experimental design and data analysis will usually depend on data from your group and all members of your lab section.

Weekly Laboratory Routine

Because of the exploratory nature of this laboratory, it is absolutely essential that you prepare for each experiment ahead of time. This is more important than in any of your previous laboratory courses. You have a limited amount of time in the laboratory to collect data each week, so you need to be ready to work when you arrive. **The experiments are designed to be completed in the 3 hours scheduled each week.** You must be prepared to begin work when you arrive in the lab to complete your work in the scheduled time. To **help you prepare**, there will be **assigned pre-lab background reading, an electronic pre-lab assignment and a laboratory discussion period** (See the CHEM 211L weekly schedule pp. 3-7). All of the assigned reading is to be **done and the electronic pre-lab assignment submitted on the course Blackboard site before 10:00 PM on the day before the pre-lab discussion.** Your lab notebook with procedure and supporting tables of compounds must be set up before you come to your afternoon lab section.

The laboratory schedule (See CHEM 211L Weekly Schedule, pp. 3-7) should provide you with sufficient time to complete required work, so the deadlines for laboratory reports are firm. At first, while you are unfamiliar with the lab and you are learning many new techniques, things will go more slowly. Work at a pace that is comfortable for you, but keep an eye on the schedule. As the semester progresses your abilities will grow. You must learn how to perform the techniques correctly and efficiently. As you become familiar with the major techniques, you will be able to work faster. You will also be able to keep several different procedures going at once. You will learn when your data are good enough to support claims and when you need to repeat a procedure that did not yield data that are reliable enough to fulfill your needs. You will find that it is more efficient to work with care and produce good results than to rush through a procedure just to get it finished. Remember, the purpose of each experiment is to answer a specific question. Be sure to ask for help if things seem confusing. You are here to learn; we do not expect you to be able to do everything perfectly at once. Enjoy yourself as you learn.

Resources

Key “tools” that will assist with pre-lab and in-lab assignments are the *Merck Index*, the Table of Physical Properties of Organic Compounds that is located in Section C of the *CRC Handbook of Chemistry and Physics* and the Aldrich Chemical Co. *Catalogue of Organic Chemicals*; they are available in the laboratory and in the Chemistry Reading Room (217 Collier). SciFinder Scholar is an on-line search engine that is dedicated to searching chemical literature. You will be introduced to it as part of your Library Project next semester. Several Internet sites provide information on structures and properties of organic molecules. Some you might find useful are: The Chemical Database (University of Akron <http://ull.chemistry.uakron.edu/erd/>), the NIST Chemistry WebBook (<http://webbook.nist.gov/chemistry/>), The Chemical Synthesis Chemical Database (www.chemsynthesis.com/), ChemSpider (<http://www.chemspider.com>), Chemical structures Project (http://chem-file.sourceforge.net/data/name_index_en.html) or About.com:Chemistry (<http://chemistry.about.com/od/chemicalstructures/a/structuresa.htm>).

Administrative Details.

Required Materials: (All available at the college bookstore)

- Lab Text: *-Making the Connections: A How-To Guide for Organic Chemistry Lab Techniques*; Second Edition—by Anne B Padías, Hayden McNeil 2011 (ISBN: 978-0-7380-4135-3- paperback) (**Padías**)
- Lab Notebook: Must have a **hard cover** and be **permanently bound** (not spiral), must also fit in the inside pocket of your data binder.
- Data Binder: A **three-ring loose-leaf binder** (1.5-2 inch) with a **hard cover**, at least **10 tab dividers** and an **inside pocket** that will accommodate your lab notebook.

Grades:

In general, satisfactory completion of all work, (prelab assignments, necessary lab work, laboratory notebook records, and laboratory reports) will earn a final grade of B for the lab. Higher grades will be given for demonstration of excellent understanding of the concepts, preparation for the lab periods, performance in the lab, neat and thorough lab notebook records, and well-written lab reports.

Your grade for the laboratory portion of the course will be calculated as indicated below:

- 30% The quality and completeness of the laboratory notebook and data binder
- 30% The quality and completeness of the laboratory reports
- 25% Performance in the laboratory
- 10% Preparation for each laboratory period (Includes completion of electronic pre-lab assignments, contributions to pre-lab discussions and initial awareness of experiment requirements.)
- 5% Attendance in the laboratory discussion (AM)

Lab Records

Notebook

This semester's laboratory provides you with experiences that are designed to help you develop some general skills. Many of the procedures you will be doing for the first time in this particular way. It is very important that you keep accurate and orderly records of your activities so that you (and we) can understand what you have done after you have completed your work. Your records must be permanent; all entries are made using **black or blue non-erasable ink**. These are the standards required in all scientific and medical work, in research, analysis, or clinic whether in corporations, academia, or private practice. (See pp. 14-16 for CHEM 211-212 notebook format.)

Data Binder

Your data binder will provide a convenient (and required) place to keep the spectra, chromatograms, and other sheets of data generated by your lab work. Organic chemistry lab creates many types of data that are not easily attached to a standard lab notebook. Your data binder will help you keep all data related to each activity well organized. Your notebook and data binder materials will be evaluated each laboratory period. (See p. 17-18 for CHEM 211-212 data binder format.)

Reasons for Keeping Well-Organized Laboratory Records (See also Padías pp. 5-12)

Laboratory work in science requires certain technical talents. However, cleverly devised and precisely executed procedures will have no impact on the scientific community if a researcher cannot communicate the results to others. All of the information in the world is useless if it cannot be retrieved conveniently. Thus, along with talent in the laboratory, a competent researcher must be able to organize, analyze and convey his or her results and conclusions to others. The basis for good scientific communication is solid data. In the world of professional research and analysis, notebooks document dates of discoveries for patent and publication purposes. So experience with keeping accurate and well-organized lab records is an important credential for a developing scientist.

There should be a natural flow to the information, and it should be blocked out in a way that is pleasing to the eye (with the idea that someone else might need it).

Typical Laboratory Records Should Have:

- Dated and timed entries
- Records in black or blue non-erasable ink
- Up-to-date "Table of Contents" with titles of all major parts of experiments
- Any references or citations that were used to prepare for and perform the procedure. These should include references to coworkers and their notebooks.
- A planned procedure written out beforehand and subsequently modified as performed.
- Any and all manipulations performed and observations that are made.
- Chromatographic data including TLC plates (in your data binder), solvents used, and any visualization methods used, GC chromatogram tracings (in your data binder) with all-important chromatographic conditions.
- Spectral traces (in your data binder) with all-important conditions for their collection.
- Mistakes crossed out with a single line and no obliterations.
- Speculations! -- These remind you of what you were thinking when the work was done.
- "Continued to page..." and "continued from page..." labels if needed.

So that you can remind yourself about your development, you may find it useful to prepare a summary for yourself at the end of an activity or experiment.

To fulfill the above requirements, any well-organized format for a laboratory notebook is sufficient. However, in a course where the work of a large number of students must be evaluated by another person, efficiency and accuracy of evaluation require a certain uniformity of format. When the evaluator knows where to find the required information she/he can focus his/her major efforts on the work and giving students suggestions on how to improve their laboratory work and records. Consequently, a specific order and placement of information in your notebook and data binder are outlined in the **Laboratory Record Format** section in Part III. By following this outline, you will assure that your work will receive maximum consideration.

Laboratory Reports

Once your laboratory work on each experiment is complete you will collaborate with your lab group mates to summarize and interpret class results in a laboratory report, which you will prepare as a group (one report per group) using a Blackboard Wiki. Specific due dates for your reports are listed in the *Laboratory Schedule* (pp. 3-7). In addition to an organized summary of your results, your laboratory report will include your claims and a complete warrant (the logic that led to your claims) with citations to specific elements of your data that support your arguments. The general format for lab reports is outlined in the **Laboratory Report Format** section in Part III. (See p. 18-19 for CHEM 211-212 data binder format.) There will be specific Laboratory Format Sheets on the course website for each experiment.

PART II: Laboratory Rules and Regulations

General Rules and Regulations

Laboratory Neatness

Neatness is essential for safety and for efficient work in the laboratory.

1. Keep the lab uncluttered by leaving book bags and all non-essential books under your lab desk (NOT in the aisle). Outer-clothing should be hung on the pegs in the hall or the front of the laboratory.
2. If you spill anything anywhere in the laboratory, clean it up immediately and leave a clean space for your neighbors. This applies particularly to the balances and melting point devices.
3. If problems arise, then each week specific students will be given responsibility for seeing that certain areas are clean and orderly at the end of the period. Please cooperate.
4. If you spill acids, bases or other corrosive chemicals, inform the instructor, neutralize as directed by the instructor and wash contaminated surfaces with copious amounts of water.

5. For reasons of safety and obtaining dependable results, all glassware must be thoroughly clean. After disposing of contents responsibly (see waste disposal instructions for each experiment), wash glassware with hot soapy water and a test tube brush. In some cases, organic compounds may not be removed by detergent and require a small acetone rinse (See Padías p. 18).

Laboratory Reagents

1. Most of the reagents used in this laboratory are irritants and/or toxic. Be careful handling reagents. Gloves are available should you wish to use them, but remember that gloves do not give you 100% protection from all lab reagents. If reagents come in contact with your skin or gloves, wash them off immediately with soap and water. It is also a good practice to wash your hands periodically to remove any material that may have been left on your hands by incidental contact. Finally, be sure to wash your hands with *soap* and *hot water* before leaving the laboratory.
2. **Use reagent bottles only in the areas where they are provided.** Solid reagents for this course will be set out on benches at the side of the lab near the balances, and occasionally under the hood. Volatile or corrosive liquid reagents will be under the hood.
3. Try to take no more of the reagents than you need.
4. Always replace reagent container caps after you obtain samples.
5. If by accident you take an excess amount of a reagent, share it with a fellow student or dispose of the excess properly. Never pour anything back into a reagent bottle!
6. Always dispose of chemicals properly.
7. In general, organic wastes are divided into two classes: "halogenated", compounds containing one or more atoms of F, Cl, Br, or I; and "non-halogenated". Halogenated compounds require special disposal because of their particular ability to harm the atmosphere.
8. Specific instructions for reagent disposal will be given for each activity; however, for our purposes, they fall into seven categories, which are listed with proper disposal methods in the Table 1 below. **Tape or glue Table 1 on the inside front cover of your lab notebook.**

Table 1: Laboratory Waste Disposal Categories

Category	Compound Type	Disposal
1	Non-halogenated organic solids	Labeled waste containers in waste hood
2	Halogenated organic solids	Labeled waste containers in waste hood
3	Water-insoluble - halogenated organic liquids	Labeled waste containers in waste hood
4	Water-insoluble non-halogenated organic liquids	Labeled waste containers in waste hood
5	Acidic or Basic aqueous solutions	Labeled waste containers in waste hood
6	Water-soluble non-halogenated organic liquids	Down lab drains with a flow of cold water
7	Inorganic solids	Varies with compound

Safety Regulations (Also read Padías pp. 1-4)

1. You may never under any circumstances work in the laboratory outside of your assigned laboratory period without permission from **Dan Libby**.
2. Your eyes must be protected by goggles at all times. If chemicals come in contact with your eyes, flush your eyes copiously with water for at least five minutes (The eye wash station will be demonstrated during the safety lecture).
3. Accidents. Report all accidents to an instructor immediately!! First aid is essential. Flush all chemical splashes with much water. Use the eye wash station for chemicals in your eyes. Use the spray attached to the eye wash sink to wash spills from your clothing immediately and wash them out liberally with water after the laboratory period.

4. Food. No food or drinks are allowed anywhere in the laboratory. If you bring a lunch, snack or drink, it must be kept in the hall outside the lab. No food consumption or gum chewing is allowed in the laboratory. Also, no make-up or lip balm is to be applied in the lab.
5. Broken Glass. Dispose of any broken glass in the glass disposal box at the back of the laboratory.
6. Broken Thermometers. Inform an instructor immediately if a thermometer is broken. Any spilled mercury must be recovered properly.
7. Fire Hazards.
 - a. The solvents used in the laboratory (alcohols, ether, petroleum ether, etc.) are highly flammable. To minimize the danger of fire, no flames are allowed in the laboratory without specific authorization.
 - b. In case of fire, remain calm! Most fires are contained in beakers or flasks and can be easily smothered by being covered with a watch glass. More extensive fires should be smothered using a carbon dioxide extinguisher. Since water does not dissolve many organic solvents, it will not extinguish most fires, but will cause them to spread. So use a laboratory fire extinguisher rather than water on a fire.
8. General Safety
 - a. **Appropriate clothing** must be worn to protect your body from chemical spills. Clothing **must have sleeves** and **cover you from your neck to your knees**. If not suitably covered, you must wear a lab coat. Failure to do so will lower your lab grade. **Closed toed shoes** are required; you will not be allowed in lab without them.
 - b. Keep long hair tied back out of the way of chemicals and equipment.
 - c. Beware of hot glassware. Do not touch it until it has had time to cool.
 - d. To insert glass tubes or thermometers through stoppers or adapters, first lubricate both the glass and the stopper with glycerin or stopcock grease. Then, using a towel, cloth or other hand protection, and holding the glass tube or thermometer near the end closer to the stopper, insert the glass tube into the stopper, a few mm at a time, using a rotating motion.

Daily Routines

Laboratory Equipment

1. Keep all metal apparatus in a dry place. Water tends to rust equipment and cause drawers to swell so that they become difficult to open. Consequently, if you spill water in a drawer, immediately take time to dry it carefully.
2. At the end of each lab day, equipment that is left around the lab will be placed in a bin at the back of the lab where it will be available for any student in the course. If you lose or break a piece of equipment check the bin for a replacement. If none is available, ask your lab assistant for a replacement. There is no charge for replacement of broken equipment. However, materials used in this laboratory are expensive so try to be careful. Also, excessive loss or breakage can be an indication of poor organization, technique or lack of preparation and can adversely affect your lab grade.

Labeling Chemical Samples

All samples that are **stored in your tote tray** or **turned in** to your instructor must be **labeled** according to the following format:

Name of Substance
Lab Notebook page
Date
Student Name
Lab Section Day

Figure 1: Sample Compound Label

Post-lab Routine

At the end of each lab period:

1. Wipe off your personal bench-top area.
2. Clear any debris from your sink.
3. Check that the common drawer and sink items are in place (see equipment lists on the next page).
4. Wash your hands.

EQUIPMENT LIST

Personal Tote Tray Contents

- | | |
|---|--|
| ☐ 5 Beakers, 100, 250, 400, 600, 1000 ml | ☐ 1 Stemless funnel |
| ☐ 1 Büchner or Hirsh funnel w/ rubber adapter | ☐ 1 Stirring rod |
| ☐ 1 Conical funnel | ☐ 1 Test tube clamp |
| ☐ 5 Droppers | ☐ 1 Test tube rack |
| ☐ 8 Erlenmeyer flasks, 50, 3x125, 2x250, 2x500 ml | ☐ 1 Test tube block |
| ☐ 1 Filter flask, 500 ml | ☐ 10 Test tubes, 10x75 mm |
| ☐ 2 Graduated cylinders: 10, 50 ml | ☐ 6 Test tubes, 13x100 mm |
| ☐ 1 Powder funnel | ☐ 6 Test tubes, 15x150 mm |
| ☐ 1 Ruler, 6 in. | ☐ 2 Test tubes, 25x175 mm |
| ☐ 1 Scupula | ☐ 3 Vials, w/ caps |
| ☐ 1 Separatory 250 ml funnel w/ stopper & stopcock | ☐ 2 Watch glasses |
| ☐ 1 Spatula | ☐ Goggles (\$5 replacement fee if lost) |
-

Common Drawer Equipment

- | | |
|--|--|
| ☐ 3 Ring stands (bench top) | ☐ 1 pH paper vial |
| ☐ 1 Current regulator | ☐ 4 Rubber tubings, including 1 w/ thick wall |
| ☐ 1 Heating well | ☐ 1 Tongs |
| ☐ 1 Iron ring | ☐ 4 Utility clamps w/ fasteners |
| ☐ 1 Jar, glass w/ screw top | ☐ 2 Wood blocks &/or cork rings |
| ☐ 1 Magnetic stirrer | ☐ 1 Wire gauze |
| ☐ 1 Mortar and pestle | ☐ 1 Thermometer, -10 -> 250° |
-

Sink Area Equipment

- | | |
|---|--|
| ☐ 2 Cleaning brushes (large and small) | ☐ 1 Acetone Wash bottle, (shared with other sink) |
| ☐ 1 H ₂ O Wash bottle | ☐ 2 Water baths |
| ☐ 1 Soap Wash bottle | ☐ 1 Roll of labeling tape |

Part III Laboratory Record & Report Formats

Laboratory Notebook Format

General Format

Write your **name** and **laboratory day** on the **front cover** of your notebook.

Number all of the pages (both left- and right-hand) at the top of the page.

Reserve the **first four numbered pages** for the **table of contents**. As the semester proceeds, enter the **Experiment number** and the **Title** of each experiment and the **Designation and Title of each Part** (E.g. Experiment 1: Part A: *What can solubility in acidic and basic aqueous solutions tell us about structures of organic molecules?* etc.) into your table of contents, along with the pages on which the experimental results are recorded. Note that the **Titles of Experiments and Parts are required** in the **table of contents**; the experiment number and part designations are not sufficient.

Laboratory Notebook Records

Diagrams and descriptions are given below to illustrate where information must be entered into your laboratory notebook. Some experiments may not require all possible sections (See individual experiment descriptions). In those cases, simply omit unnecessary parts, but keep the others in the order and position (left- or right-hand page) indicated below.

<i>Left-hand Page</i>		<i>Right-hand Page</i>	
p. #			p. #
Experiment number & Title of the Experiment		Pre-lab Notes	Date
Group Name		Newly developed Claims	
Name of Groupmate(s)		Warrants for New claims	
Objectives:		General Experimental Plan	
Question of the Week			
Initial Claims		<i>Always go to next pair of clear facing pages to start your Table of Compounds and Waste Disposal Instructions.</i>	
Supporting evidence			
Warrants			
<i>Continues on the right-hand page</i>			

Figure 2. First Set of Pages of an Experiment (Always starts on a clear set of left- and right-hand pages)

Note: For experiments with more than one part (As with Experiments 1, 2 & 7), Part B is started on a new set of pages with the title of both the experiment and the part at the top of the left-hand page followed by only the QOW and pre-lab notes.

Title of the Experiment (Figure 2)

Each new experiment should always begin on a **clean pair of facing left- and right-hand pages**. The experiment number and title of the experiment are at the top of the left-hand page. Experiment numbers are not sufficient.

Group Name: (Figure 2)

Determined by group members.

Names of Groupmates: (Figure 2)

When you will be **working with labmates**, enter their **full names** below the title of the experiment or part.

Objectives: (Figure 2)

Each experiment has one or more objectives. They should be enumerated in this section, which is placed at the top of the first left-hand page following the pre-lab notes.

Question of the Week (QOW) (Figure 2)

Each week we will start with a question that will be explored in the pre-lab discussion; enter the QOW on the left-hand page below your groupmates' names. Under the QOW enter the initial claims, warrants and supporting evidence you develop **before** coming to the **AM pre-lab discussion period** for each experiment.

Pre-lab Notes (Figure 2)

This section includes your notes from the AM pre-lab discussion. It should include any new claims and warrants developed in the discussion as well as the experimental plan that arises from the discussion and **be dated to reflect the day of the discussion**. Begin this section below the QOW on the left-hand page and continue your notes on both left- and right-hand pages as necessary.

<i>Left-hand Page</i>				<i>Right-hand Page</i>																					
p. #				p. #																					
Table of Compounds <table border="1" style="width: 100%; border-collapse: collapse; margin-top: 5px;"> <thead> <tr> <th style="width: 25%;"></th> <th style="width: 25%;">mp</th> <th style="width: 25%;">bp</th> <th style="width: 25%;">density</th> </tr> </thead> <tbody> <tr><td> </td><td> </td><td> </td><td> </td></tr> <tr><td> </td><td> </td><td> </td><td> </td></tr> <tr><td> </td><td> </td><td> </td><td> </td></tr> <tr><td> </td><td> </td><td> </td><td> </td></tr> </tbody> </table>					mp	bp	density																	Waste Disposal Instructions Safety Matters: Chemical Equations <i>Always go to next pair of clear facing pages to start Procedures and Results pages.</i>	
	mp	bp	density																						

Figure 3. Next New Set of Pages of an Experiment (always starts on next clear set of left- and right-hand pages after the pre-lab notes)

Table of Compounds: (Figure 3)

This is a list of names as well as important physical (mp or bp, density, solubility in acid, base and common organic compounds) and chemical properties (acidity, basicity or flammability) of the compounds used in the experiment. When required for an experiment, this table is at the top of the left-hand page. For easy reference during the lab period, the **table must be in your notebook** rather than your data binder. So, unless otherwise directed, **always put the Table of Compounds in your notebook**

Waste Disposal Instructions: (Figure 3)

Most of the compounds that you will encounter in this course are irritants or toxic. To avoid polluting you, your classmates, and our environment, we must be careful to dispose of chemical waste properly. Thus, each experiment will provide instructions for disposal of excess reagents. These instructions are written in your notebook, at the top of the right-hand page opposite the objectives. Follow these instructions carefully.

Safety Matters: (Figure 3)

Each experiment lists a series of Safety Matters to be followed to assure safe operation in the lab. They should be enumerated in this section that immediately follows the Waste Disposal Instructions.

Chemical Equations: (Figure 3)

When experiments involve chemical reactions, **chemical** equations for the reactions are written **using structural formulas of the organic compounds**. Equations are placed on the right-hand page below the waste safety matters.

Procedure (Always starts on next clear set of left- and right-hand pages): (Figure 4)

Organized to facilitate your work in the lab. Procedure consists of sequentially numbered steps (1, 2, 3, etc.) to be carried out in the laboratory. These steps are concise statements, in your own words rather than a copy of the procedure handout for the experiment. You are required to **prepare your procedure after the pre-lab discussion** and **BEFORE you start work** in your afternoon laboratory section. This "processing" of the procedure will cause

you to think through what is planned for the afternoon and prepare you to begin work immediately upon entering your lab section.

Begin the Procedure at the top of the next available **left-hand** page after the Waste Disposal Instructions or Chemical Equations and **separate the steps by at least one line** to allow for any modifications that might be necessary as you work in the laboratory.

NOTE: When using a procedure for the first time (e.g. distillation), include the complete procedure with **diagrams of any apparatus**. When you repeat the same procedure in a later experiment, simply reference (with experiment and notebook page number) the original procedure.

<i>Left-hand Page</i>		<i>Right-hand Page</i>	
p. 35		date	p. 36
		<i>Step #'s must be opposite corresponding procedure steps.</i>	
Procedure		Results	
1. Obtain 2.2 g A.		1. 2.351 g A	
<i>space between steps</i>		<i>space between steps</i>	
2. Add A to 25 mL 2 M HCl.		2. White gas and fine pink ppt. form	
3. Etc.		3. Etc.	
4. Obtain IR spectrum of X		4. Spectrum of X can be found in my data binder on p. 1 of the Experiment 3 tab.	
↓ <i>Continues on next left-hand page.</i>		↓ <i>Continues on next right-hand page.</i>	

Figure 4. Results (always starts on the right-hand page opposite the procedure steps)

Consist of observations that may be used as indications that an experiment is proceeding properly or data collected as the result of a procedural step. Observations such as colors of solids or liquids, color changes, pH, evolution of gas and/or heat, violent reactions, dissolution of a solid, etc. and data such as wts., volumes, mp's, bp's, etc. are recorded in your notebook Results section as the work proceeds.

Each entry in your Results section is directly opposite the procedure step to which it refers and be designated with the number of that step. (As illustrated in the diagram above) When data cannot be conveniently attached to the results section, it is **kept in the appropriate section** of your data binder and a **note to "see data binder - Experiment # and page #"** are included at the appropriate position in your results section. (See the Data Binder section below).

<i>Left-hand Page</i>		<i>Right-hand Page</i>	
p. #		date	p. #
Reflections		Reflections (Continued)	
↓ <i>Continues on next right-hand page.</i>		↓ <i>Continues on next left-hand page.</i>	

Figure 5. Next New Set of Pages of an Experiment (always starts on the first left-hand page following Procedure and Results)

Reflections (always starts on the first left-hand page following Procedure and Results): (Figure 5)

This section is optional in your notebook and is designed for you to note your thinking soon after the your lab period. It should include any insights that you believe may be important for you to consider in analyzing your data and preparing your lab report.

Data Binder Format

General Description

Your data binder needs to be a 3-ring, loose-leaf notebook binder (1.5-2 inch). It must have a pocket on the inside of its front cover sufficient to accommodate your lab notebook and contain at least 9 tab dividers that separate the sections described below. Your data binder will be used to store data (e.g. thin-layer chromatography plates, infrared, mass and NMR spectra, etc.) that don't fit conveniently into your notebook. Also place returned lab reports in their appropriate section.

General Format

Write your **name** and **laboratory day** on the **front cover** and on **the spine** of your data binder. (lab label tape works for this).

The first item in the data binder is this lab manual. Sections following the lab manual are separated by tabs, which are **labeled as indicated below**. Each experiment section includes all special handout (procedure, in-lab questions, lab report instructions, etc.) and any data sheets that are not easily incorporated in your notebook. Materials to be included in each section must be sequentially numbered and have 3-holes that match the rings of the binder or be permanently attached to a sheet that has the appropriate holes.

Data Binder Sections

Lab Manual

Weekly Post-lab Notebook Checklists:

Experiments 1

Experiments 2

Experiments 3

Experiments 4

Experiments 5

Experiments 6

Experiments 7

Experiments 8

Labeling and Cross Referencing Data:

Each piece of data placed in your binder must be **labeled with your name, a descriptive title of the data and a reference to the page of your notebook and number of the procedure step** that produced the item. Descriptions must be sufficient to identify the data without reference to the notebook. (Figure 6.)

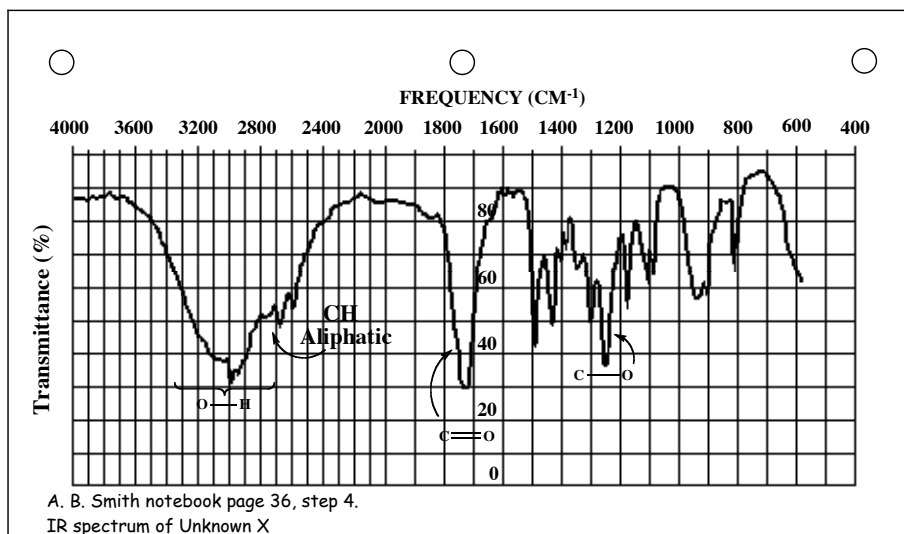


Figure 6. Sample Data Binder Sheet

In addition, your **notebook results section should have an entry to indicate that the data from a procedure step is in your data binder.** (e.g. For the spectrum shown in Figure 6, is cited (Figure 4) on the results page across from step 4 on p. 35 of the notebook, "Spectrum of X can be found in my data binder on p. 1 of the Experiment 3 tab."

Laboratory Report Format

General Description

After completing all activities in an experiment you will collaborate with your groupmates to compile and analyze your data and data from your lab section. You will then **use a Blackboard group wiki to prepare and submit a group lab report** on-line. Your analyses will usually be assisted by in-lab and post-lab questions distributed for most experiments. All reports are to be prepared and submitted through the appropriate Blackboard group wiki.

Required Sections – Each section will be on a separate wiki page:

(Specific descriptions for sections of each experimental report (Lab Report Format Sheets) will be provided on the experiment page of the course website.

Title Page-First page of the report wiki:

Description
In a **vertical list centered near the top** of the page:
your **lab day and section**
your **lab group name**
complete names of all group members
date the report is **submitted**.
Centered both vertically and horizontally on the page
The **experiment number** and **title of the experiment**
See the Figure 7.

Figure 7. Title page Template

CHEM 211L Tuesday- Section A
The Distillers
James Johnson Cheryl Smith James Walker Mary Yaro
September 4, 2008
 Experiment 1: How Can Representations of Molecular Structures Help Us Find All of the Isomers for Any Molecular Formula?

The Question of the Week – Second page of the report wiki:

In this section, state The Question of the Week and then provide a brief (50 words or less) statement of your initial claims concerning the QOW and your warrants explaining how your claims were derived.

Experimental Plan - Third page of the report wiki:

Then briefly describe (50 words or less) the experimental plan to answer to the QOW. There should also be some discussion of how the experimental plan was developed during your pre-lab work. This is a general plan and should not contain details of materials used.

NOTE: Reports for experiments with two parts, will have two QOW and Experimental Plan sections.

Summary of Results - Fourth page of the report wiki: (See also Collaboration and Academic Honesty p.20):

This section contains **only experimental data organized to facilitate citing the data** in the Claims, Warrants and Backing section. The only text in this section is in the references to sources of the data. **BEFORE** preparing your Summary of Results, you **must analyze all data** and **determine your final claims concerning the QOW** for the experiment. This analysis will inform your choices of method for displaying and organizing the data.

Figure 8: Advice on Tables, Graphs and Diagrams

Required characteristics:

Each item in this section has:

A **designation** (e.g. Table 1, Figure 1, etc.) to facilitate citing in your discussion.

A **descriptive title** to indicate what data is being presented (e.g. Distillers' Group Boiling Points).

Specific references to laboratory notebook and/or data binder pages containing the original data. (Indicate both the group member's name and numbers of notebook pages containing the data.)

Mechanics:

If you need assistance with inserting tables or graphs or other word processing techniques in the wiki, consult your instructor **before submitting** your report.

Claims, Warrants and Backing – Fifth page of the report wiki: (Not more than 300 words total)

In this section state each of **your final claims** concerning answers to the Question of the Week followed by your warrant for that claim and any backing that supports your warrant. Your warrant shows how your claim was derived from the data and cites the **specific data entries in the Summary of Results section** that form **the basis for your claim**. **Your warrant** presents the reasoning process that allowed **you** to discern your claims from the cited data. When appropriate, you should also include **theoretical backing that reinforces your warrant**.

For experiments that lead to more than one claim, provide each set of claim, warrant and backing in sequence.

Reflections – Sixth page of the report wiki: (Not more than 150 words)

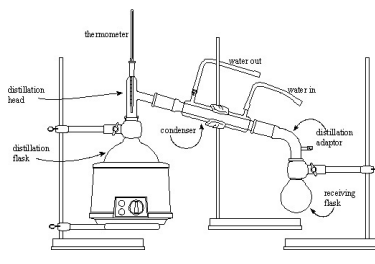
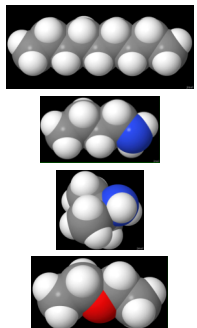
This section provides an opportunity for you to look back on your experience in the experiment and analyze how it has affected you. What new insights did you have and how has your experience changed or reinforced your understanding of chemical concepts? Finally, briefly explain your current understanding of the important concepts developed or supported by your experience and provide warrants to justify your positions.

Collaboration and Academic Honesty

Collaboration among students in collecting data in the lab is required. It is also encouraged in the process of devising claims and preparing Results sections of lab reports. So **all members of a group may use the same claims and Results section** for their lab reports, but **all other sections of the report should original and represent individual student understanding** after group discussions of the material. Educational research indicates that students learn best when they engage in discussions and analyses of information with their peers.

To be fair to all students in the course and to assure maximum learning for each student, we follow all the guidelines for academic honesty spelled out in the *Moravian College Student Handbook* (See College Website <http://www.moravian.edu/studentLife/handbook/academic/academic2.html>).

Part IV. Experiments**Experiment 1
Part A**

	<i>How is the Boiling Behavior of a Liquid Related to Its Molecular Structure and Purity?</i>	
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A. Pre-lab Preparation Assignment:

1. Read: Experiment 1, pp. 20-22.
2. Read: -Basic Laboratory Techniques (Padías pp. 16-21)
-Heating Methods (Padías pp. 22-24)
-Simple Distillation (Padías pp. 141-143)
-Boiling Points (Padías p. 53)
3. Complete the Prelab activity on the course website by **10:00 PM, Thursday, August 29** on the **Course Blackboard Site** as explained on the Course Information page.
4. Consider possible initial claims and warrants concerning the QOW (See below)
5. In your lab notebook: (See Lab Manual pp. 14-16 for format.)
 - a. Create a Table on Contents and enter the Experiment number, title and beginning page for Experiment 1.
 - b. Number all pages.
 - c. Enter the title of the experiment, your group name, names of group members, objectives of the experiment and QOW on the appropriate first left-hand page.
 - d. Enter your initial claims and warrants concerning the QOW. (See section D on the next page.)
 - e. Create a Pre-Lab Notes section.
6. Bring your notebook with your initial claims and warrants concerning the QOW into the AM lab discussion period on Friday, August 30.

B. Objectives:

1. To interpret the effects of observable molecular characteristics on boiling points of liquids in terms changes in the strengths of intermolecular forces.
2. To explore how the various types of intermolecular forces exhibited by a molecule work together to determine its boiling point.
3. To master distillation as a technique for purifying liquids and determining their boiling points.

C. Introduction:

In class you have gained experience with comparing written molecular structures to determine if they are the same or different. In those activities you worked with representations of the structures of compounds. In the laboratory it isn't possible to see the structure of a substance. So chemists define substances on the basis of an accumulation of observable properties. For example, when we say "water," we mean "that clear, colorless,

odorless liquid with a boiling point of 100°C, freezing point of 0°C, a density of 1 g/ml that dissolves substances like salt, that upon electrolysis gives a mixture of hydrogen and oxygen gases in a definite ratio"...and so forth. Using our molecular metaphor of matter we interpret the properties of "water" in terms of a molecular structure, H-O-H. Thus to identify a compound, it is critical to be able to determine and compare the physical properties of substances accurately and reproducibly.

Experiment 1 introduces you to a simple but important measurement technique for observing the boiling behavior of a liquid sample to judge its purity and determine the compound's boiling point. The boiling point of a compound is the temperature at which its liquid and vapor forms are at equilibrium. At this temperature, bubbles form in the liquid so this characteristic temperature for a substance is called its boiling point (bp). Molecules must move apart from each other as they pass from the liquid phase to the gas phase. In the gas phase the molecules are moving more randomly, so the entropy change from liquid to gas is favorable. However, the intermolecular forces in the liquid must be overcome and that process is unfavorable; it requires energy. In determining the boiling point of a liquid we supply the needed energy as heat. Since the entropy change in moving molecules from the liquid phase to gas phase is similar for most organic molecules, differences in boiling points for different liquids must result from differences in the strengths of intermolecular forces in the liquid. So, we can use the boiling point of a liquid as a measure of the strengths of the intermolecular forces among molecules in the liquid phase. The higher the boiling point the stronger the intermolecular forces. In earlier courses you have learned about major types of intermolecular forces, London Forces, dipole-dipole forces and hydrogen bonding, and their relative strengths. But until now you haven't had to consider how the different types of intermolecular forces work together to determine the overall intermolecular forces among molecules of a compound.

This experiment explores the relationships between the bps (strengths of intermolecular forces) of a group of liquids and their structures. There is a great variety of ways in which atoms can be connected to make different molecules and these different molecules, even if they have the same molecular formula, can have many different characteristics. If we extend our comparisons to include molecules with different molecular formulas, the number of variables in their potential structures increases greatly. Our Question of the Week leads us to consider potential structural characteristics that might affect the strengths of intermolecular forces of the liquid form of a substance and how they work together to control the bp.

D. Question of the Week:

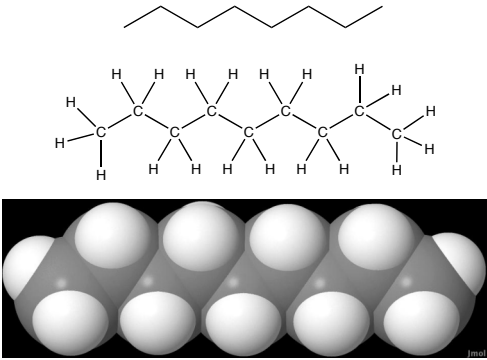
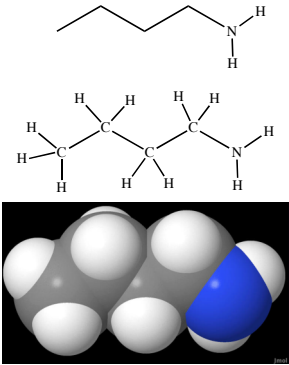
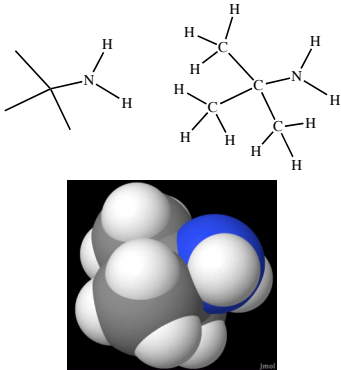
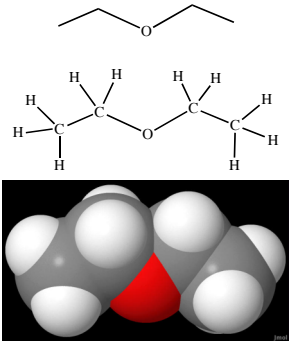
How does overall molecular structure determine the boiling point of a compound?

To provide some context for our consideration of the QOW, multiple representations of a few molecules along with their molecular formulas, molecular masses and bps are presented in Table 1. The bond-line and dash representations show the atoms in the molecules, but the third representation (often called spacefilling) also shows the surface of the electron density of the atoms in the molecule with atoms indicated only by color (white=Hydrogen, gray=carbon, blue=nitrogen and red=oxygen – you can see color representations on the Experiment 1 page of the course website). You can think of spacefilling representations as showing the surface of the molecule available to interact with other molecules in a sample of the substance.

Initial Claims and Warrants

Consider the structural characteristics of the four molecules provided on the next page and propose two structural differences that might cause the observed differences in boiling points. These are your initial claims. Support your claims with your warrant, the logic you used to create the claim. Enter your initial into your notebook as described in part A.

Table 1: Some characteristics of representative liquids.

 Nonane C_9H_{20} Mol Mass = 128 Bp = 151 °C	 1-Butanamine $C_4H_{11}N$ Mol Mass = 73 Bp = 78 °C
 2-Methyl-2-propanamine $C_4H_{11}N$ Mol Mass = 73 Bp = 46 °C	 Diethyl ether $C_4H_{10}O$ Mol Mass = 74 Bp = 35 °C

E. Key Terms/concepts/techniques:

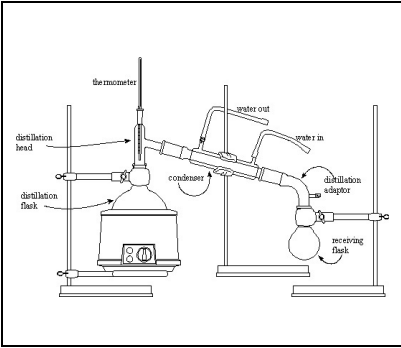
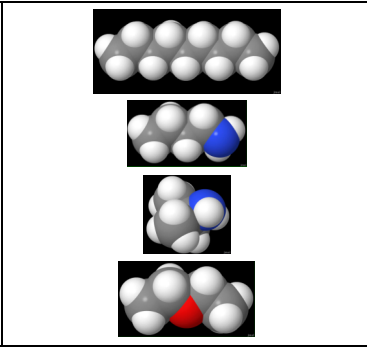
1. States of matter
2. Phase changes
3. Isomers
4. Boiling point
5. Intermolecular forces

F. Safety Matters:

1. Safety goggles are required.
2. Shoes that cover the tops of feet.
3. Clothing that covers from neck to knees.
4. Flammable liquids-no flames in the lab.

Experiment 1

Part B

	<i>How is the Boiling Behavior of a Liquid Related to Its Molecule Structure and Purity?</i>	
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A. Pre-lab Preparation Assignment:

1. Read: Experiment 1 part B, pp. 23-24.
2. Read: -Chromatography (Padías p. 162)
-Gas Chromatography (Padías pp. 179-180)
3. Complete the Prelab activity on the course website by **10:00 PM, Thursday, September 5** on the **Course Blackboard Site** as explained on the Course Information page.
4. Consider possible initial claims and warrants concerning the QOW (See below)
5. In your lab notebook: (See Lab Manual pp. 14-16 for format.)
 - a. Enter the title of the experiment, your group name, names of group members and QOW on the appropriate first left-hand page.
 - b. Enter your initial claims and warrants concerning the QOW. (See section D below.)
 - c. Create a Pre-Lab Notes section.
 - d. Up-date the Table of Contents.
6. Bring your notebook with your initial claims and warrants concerning the QOW into the AM lab discussion period on Friday, September 6.

B. Objectives:

1. To master gas chromatography as a technique for analyzing liquid samples to determine their purity.
2. To relate the distillation behavior of a liquid sample to its purity.

C. Introduction:

In Part A of this experiment, we assumed that liquids that displayed nearly constant vapor temperature during a distillation consisted of single compound. In Part B we analyze our distilled samples to actually determine how many compounds are present. We will use gas chromatography, a standard method for separating liquid sample into their component compounds and determining the relative amounts of each component.

D. Question of the Week:

How can we determine the purity of a liquid sample?

Initial Claims and Warrants

Based on your reading about gas chromatography, propose what data will be acquired if your distilled sample contains a single compound. How will the data differ if your sample contains two or three compounds? How will you be able to determine if your distilled sample is pure? Support your claims with your warrant used to create them. Enter them into your notebook before coming to the AM lab discussion.

E. Key Terms/concepts/techniques:

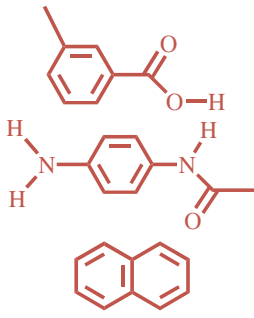
1. States of matter
2. Phase changes
3. Isomers
4. Boiling point
5. Intermolecular forces
6. Chromatography
7. Gas Chromatography

F. Safety Matters:

1. Safety goggles are required.
2. Shoes that cover the tops of feet.
3. Clothing that covers from neck to knees.
4. Flammable liquids-no flames in the lab.

Experiment 2

Part A

 Solutes	<i>How Does the pH of Aqueous Solutions Affect the Solubilities of Organic Compounds?</i>	H_3O^+ Cl^- Na^+ OH^- H_2O Solvents
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A. Pre-lab Preparation Assignment:

1. Read: -Experiment 2 Part A, pp. 25-26 and Appendix A1.
2. Complete the electronic Prelab Questions (course website) by **10:00 PM, Sunday, September 15** on the **Course Blackboard Site** as explained on the Course Information page.
3. Consider possible initial claims and warrants for the QOW (See below)
4. In your laboratory notebook: (See Lab Manual pp. 14-16 for format.)
 - a. Enter the title of this experiment and Part, your group name, names of group members, objective of the experiment and QOW on the appropriate first left-hand page.
 - b. Enter your initial claims and warrants concerning the QOW. (See section D below.)
 - c. Create a Pre-Lab Notes section
 - d. Up-date the Table of Contents.
5. Bring your notebook and your initial claims and warrants concerning the QOW to the AM lab discussion period on Monday, September 16.

B. Objectives:

1. To determine how solubilities of organic compounds do or do not change when the pH of the aqueous solvent changes.
2. To develop a working model of how pH effects on solubility of organic compounds relate to the solute structure.
3. To relate acidic and basic properties of organic compounds to their structures.
4. To explore possible application of pH effects for separating mixtures of compounds.

C. Introduction:

In this experiment, we will look at solubility of organic compounds in water and how changes in the pH of aqueous media might or might not affect the solubilities of various types of organic compounds.

D. Question of the Week:

What can solubility in acidic and basic aqueous solutions tell us about structures of organic molecules?

Appendix A presents the general effects of the pH of aqueous solutions on acidic, basic and neutral compounds. So by comparing the solubilities of a compound in water with its solubility in aqueous NaOH solution we can determine if the compound is acidic. Similarly, by comparing the solubilities of a compound in water with its solubility in aqueous HCl solution we can determine if the compound is basic.

Initial Claims and Warrants

After examining Appendix A carefully:

1. Describe the change in solubility of a compound between water and aqueous HCl solution that would indicate that the compound is basic. Provide your warrant.
2. Describe the change in solubility of a compound between water and aqueous NaOH solution would indicate that the compound is acidic. Provide your warrant.
3. Finally describe the kind of data you would need to link the acidity or basicity of a compound to a particular atom or group of atom in its structure. Provide your warrant.

E. Key Terms/concepts/techniques:

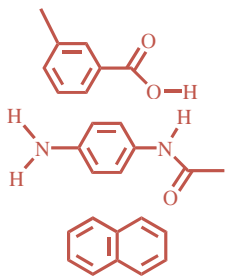
1. pH
2. Composition of acidic and basic aqueous solutions.
3. Proton transfer reactions
4. Relative solubilities of neutral and ionic compounds.
5. Acidic and basic functional groups.

F. Safety Matters:

1. Safety goggles are required.
2. Shoes must cover the tops of your feet.
3. Clothing that covers from neck to knees.
4. Avoid skin contact with organic compounds and acidic and basic solutions; wash your hands periodically during the laboratory period.

Experiment 2

Part B

 Solutes	<i>How Does the pH of Aqueous Solutions Affect the Solubilities of Organic Compounds?</i>	H_3O^+ Cl^- Na^+ OH^- H_2O Solvents
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A. Pre-lab Preparation Assignment:

1. Read: -Experiment 2 Part B, pp. 27-28 and Review Appendix A1.
2. Complete work on Part A including IN-Lab and POST-Lab questions. (See website Experiment 2 Part A page.)
3. Complete the electronic Prelab Questions (course website) and submit your answers by **10:00 PM Sunday, September 22** on the **Course Blackboard Site** as explained on the Course Information page.
4. Consider possible initial claims and warrants for the QOW (See below)
5. In your laboratory notebook: (See Lab Manual pp. 14-16 for format.)
 - a. Enter the title of Part B, objectives of the experiment and QOW on the appropriate first left-hand page.
 - b. Enter your initial claims and warrants concerning the QOW.
 - c. Create a Pre-Lab Notes section.
 - d. Up-date the Table of Contents.
6. Bring your notebook and your initial claims and warrants concerning the QOW to the AM lab discussion period on Friday, October 7.

B. Objectives:

1. To develop separation methods based on differences in solute solubilities in acid and/or basic aqueous solutions.
2. To successfully separate a mixture of two compounds and isolate both components of the mixture.

C. Introduction:

In Part A, we explored the relationships between solute molecular structure and pH effects on solubility in water. This week we explore how solubility in acidic and basic solution can be used to separate mixtures of compounds.

D. Question of the Week:***How can we use pH effects on solubility to separate mixtures of compounds?***

In Experiment 2 Part A, we discovered some acidic and basic functional groups and how pH changes can increase and decrease solubilities of some compounds and not others. This part of Experiment 2 proposes exploiting the behavior observed last week to separate mixtures of two compounds.

Initial Claims and Warrants

1. What kinds of compounds might be separated based on the pH effects on their solubilities? Assume that you have two compounds mixed together (a binary mixture.) Provide your warrant.
2. Propose a process by which the compounds in the binary mixture above might be separated based on the difference in solubility.

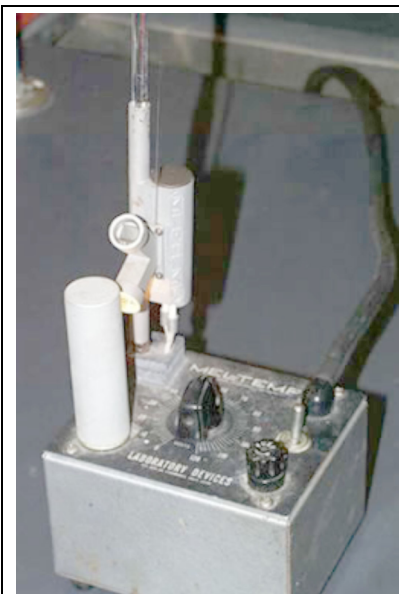
E. Key Terms/concepts/techniques:

1. Miscibility
2. Extraction

F. Safety Matters:

1. Safety goggles are required.
2. Shoes must cover the tops of your feet.
3. Clothing that covers from neck to knees.
4. Avoid skin contact with organic compounds and acidic and basic solutions; wash your hands periodically during the laboratory period.

Experiment 3



Meltemp Apparatus

How is the Melting Behavior of a Mixture of Solids Related to those of the Separate Pure Solids used to create the mixture?

A. Pre-lab Preparation Assignment:

1. Read: Experiment 3, pp. 29-30.
2. Read: Melting Point - How is it done in the lab. (Padías pp. 49-50) we will use the Mel-Temp apparatus shown in Figure 2-4, p. 52)
3. Work through the material and links on the Experiment 3 page of the course website.
4. Complete the electronic Prelab Questions (course website) by **10:00 PM, Sunday, September 29** on the **Course Blackboard Site** as explained on the Course Information page.
5. Consider possible initial claims and warrants for the QOW (See below)
6. In your laboratory notebook: (See Lab Manual pp. 14-16 for format.)
 - a. Enter the title of the experiment, your group name, names of group members, the objective of the experiment and QOW on the appropriate first left-hand page.
 - b. Enter your initial claims and warrants concerning the QOW.
 - c. Create a Pre-Lab Notes section.
 - d. Update the table of contents.
7. Bring your notebook and your initial claims and warrants concerning the QOW to the AM Lab Discussion period on Monday, September 30.

B. Objectives:

1. To master the technique for measuring melting points in capillary tubes using the Meltemp apparatus.
2. To determine the similarities and differences in melting behavior between pure solids and mixtures of solids.
3. Consider what the melting point behavior of a sample of an unknown solid might suggest about the composition of the sample.

C. Introduction:

In Experiment 1 we explored the boiling behavior of liquid compounds. We noted that the boiling point is a characteristic of a pure liquid and can assist in identifying a solid. The corresponding characteristic of a solid substance is the melting point, the temperature at which its solid and liquid forms are at equilibrium. Experiment 3 introduces you to a simple but important measurement technique (The Meltemp device) for observing the melting behavior of a solid sample and determining its melting point. You will use the technique to explore how one substance may or may not affect the melting behavior of another when they are mixed.

D. Question of the Week:

How is the melting behavior of a mixture of solids related to those of the separate pure solids used to create the mixture?

Initial Claims and Warrants

View the pre-lab clip of a pure compound melting in a capillary tube. Then consider:

1. How you would expect a mixture of two different compounds with mps of 50°C and 150°C respectively to behave when heated in a similar Meltemp device. Provide your warrant.
2. How you would expect the melting behavior of a mixture of two different compounds with mps of 95°C and 105°C compare with that of the first mixture? Provide your warrant.

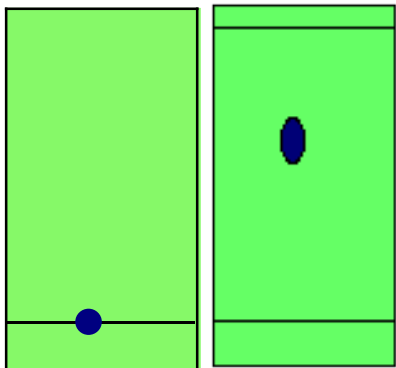
E. Safety Matters:

1. Safety goggles are required.
2. Shoes must cover the tops of your feet.
3. Clothing that covers from neck to knees.
4. Avoid skin contact with solid compounds and wash your hands periodically during the laboratory period.

F. Key Terms/concepts/techniques:

1. The Melting Point.
2. The Meltemp Apparatus.

Experiment 4

 TLC CHROMATOGRAPHY	<i>How Can We Judge The Purity of a Sample of an Unknown Solid Compound?</i>	
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A. Pre-lab Preparation Assignment:

1. Read: Experiment 4, pp. 31-32.
2. Read: Chromatography (Padías p. 162-163)
Thin-layer chromatography (Padías pp. 165-171)
3. Work through the material and links on the Experiment 3 page of the course website.
4. Complete the electronic Prelab Questions (course website) by **10:00 PM, Sunday, October 6** on the **Course Blackboard Site** as explained on the Course Information page.
5. Consider possible initial claims and warrants relative to the QOW (See below)
6. In your lab notebook: (See Lab Manual pp. 14-16 for format.)
 - a. Enter the title of the experiment, your group name, names of your group members, objectives for the experiment and QOW on the appropriate first left-hand page.
 - b. Enter your initial claims and warrants concerning the QOW.
 - c. Create a Pre-Lab Notes section.
 - d. Up-date the Table of Contents.
7. Bring your notebook and your initial claims and warrants concerning the QOW to the AM lab discussion on Friday, October 5.

B. Objectives:

1. To review the use of melting point as a measure of purity of a solid substance.
2. To master thin layer chromatography techniques for separating and analyzing solid organic samples.
3. To use thin-layer chromatography to determine the number of components in a mixture.

C. Introduction:

In the Experiment 3 you developed an understanding of the effects of mixing compounds on the melting behavior of a solid sample and in Experiment 1 you worked with gas chromatography to determine the purity of liquid samples. In Experiment 4 you will apply your understanding of the relationship of melting behavior of a solid sample to its purity and work with a new technique, thin-layer chromatography, to determine the purity of solid substances.

The reading in Padías provides background with thin-layer chromatography (TLC). Note that we will be using TLC plates coated with silica gel; they provide a very polar stationary phase. With silica gel plates, more polar molecules move more slowly than do less polar ones.

D. Question of the Week:***How can we judge the purity of an unknown solid sample?***

In formulating your initial claims consider both your experience in Experiments 1 & 4 and the introductory material on thin-layer chromatography in Padías.

Initial Claims and Warrants

Propose two analyses that could be used to judge the purity of a solid sample. Be sure to document the warrant and data (evidence) that led you to your claims.

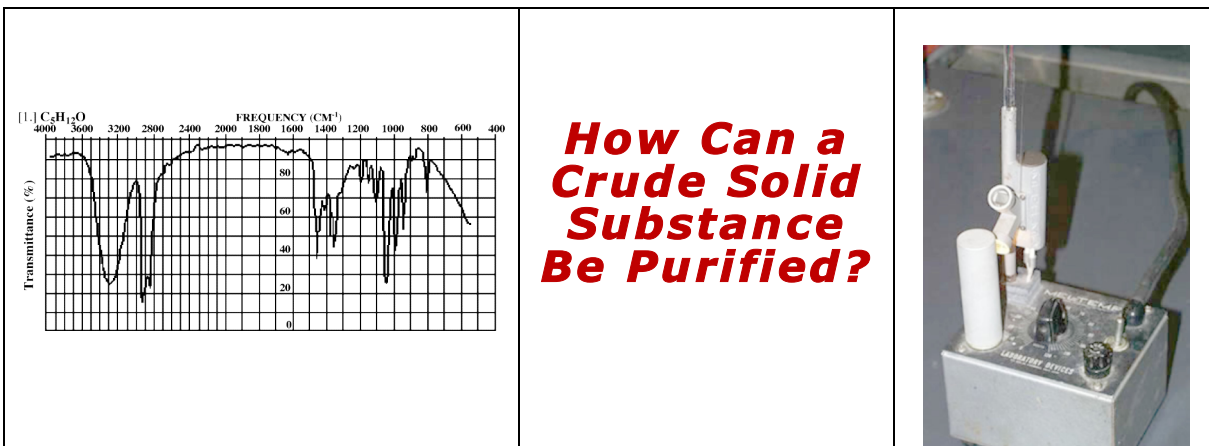
E. Key Terms/concepts/techniques:**Thin Layer chromatography**

1. Ascending vs. descending chromatography
2. Capillary action
3. Application vs. development vs. visualization vs. identification in TLC
4. R_f values

F. Safety Matters:

- a. Safety goggles are required.
- b. Shoes that cover the tops of feet.
- c. Clothing that covers from neck to knees.
- d. Flammable liquids-no flames in the lab.

Experiment 5



A. Pre-lab Preparation Assignment:

1. Read: Experiment 5, pp. 33-34.
2. Read: Vacuum Filtration (Padías p. 26-28)
3. Work through the material and link on the Experiment 5 page of the course website.
4. Complete the electronic Prelab Questions (course website) by **10:00 PM, Thursday, October 10** on the **Course Blackboard Site** as explained on the Course Information page.
5. Consider possible initial claims and warrants relative to the QOW (See below)
6. In your lab notebook: (See Lab Manual pp. 14-16 for format.)
 - a. Enter the title of the experiment, your group name, names of your group members, objectives for the experiment and QOW on the appropriate first left-hand page.
 - b. Enter your initial claims and warrants concerning the QOW.
 - c. Create a Pre-Lab Notes section.
 - d. Up-date the Table of Contents.
7. Bring your notebook and your initial claims and warrants concerning the QOW to the AM Lab Discussion period on **Friday, October 11**.

B. Objective:

1. To gain experience with selecting suitable purification reagents for an unknown organic solid.
2. To master the technique for final purification of solid organic samples.

C. Introduction:

In the experiments over the last few weeks, you have developed an understanding of the relationships between molecular structure, intermolecular forces and the effects of intermolecular forces on physical properties of organic compounds. (BP, MP, solubility) In Experiment 4 you received a sample of an unknown substance. Your analysis at that time indicated that the sample contained more than one compound. In Experiments 6 & 7 you will work on identifying your unknown compound.

As you have already seen, impurities in a solid sample can distort the properties of the major component. So, your goal in Experiment 5 is to purify your compound so that you can be sure that the observed properties are those of the compound and not distorted by impurities. In Experiment 2 you investigated extraction as a purification process and in experiment 4 you explored chromatography as a method for separating components of a mixture of solid compounds. Both of these techniques can remove major impurities, but they are not effective enough to yield pure samples. In Experiment 5 you will explore a technique that is usually the final step in the purification of a solid compound.

D. Question of the Week:***How can a crude solid substance be purified?***

In developing claims and warrants for this QOW, consider the discussions we have had concerning separating components of mixtures. As indicated above, this is the final step in purification, so the “crude sample” is usually made up of at least 80 % of the major component. If a sample is less pure than that it is usually partially purified by chromatography or extraction before the final purification.

Initial Claims and Warrants

1. How can molecules of individual components be liberated from the solid?
2. How could molecules of the major component be separately brought back to produce a purified solid sample?

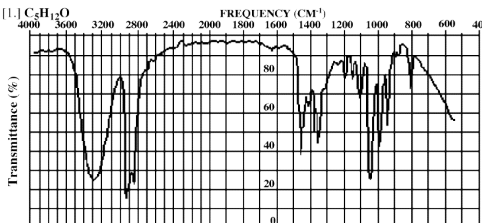
E. Key Terms/concepts/techniques:

1. Solubility
2. Recrystallization

F. Safety Matters:

- a. Safety goggles are required.
- b. Shoes that cover the tops of feet.
- c. Clothing that covers from neck to knees.
- d. Flammable liquids-no flames in the lab.

Experiment 6

	<i>How Can We Tell if Two Solid Compounds Are the Same or Different?</i>	
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A. Pre-lab Preparation Assignment:

1. Read: Experiment 6, pp. 35-36.
2. Read: Classification of organic compounds from their solubilities in aqueous media:
Lab Manual Appendix p. A1
Infrared spectroscopy:
Lab Manual Appendix p. C1
Padías pp. 66-68.
3. Work through the Infrared Spectroscopy link on the Experiment 6 page of the course website.
4. Complete the electronic Prelab Questions (course website) by **10:00 PM, Monday, October 21** on the **Course Blackboard Site** as explained on the Course Information page.
5. Consider possible claims and warrants relative to the QOW (See below)
6. In your lab notebook: (See Lab Manual pp. 14-16 for format.)
 - a. Enter the title of Experiment 6, your group name, names of your group members, objectives for the experiment and the QOW on the appropriate first left-hand page
 - b. Enter your initial claims and warrants concerning the QOW.
 - c. Create a Pre-Lab Notes section.
 - d. Up-date the Table of Contents.
7. Bring your notebook and your initial claims and warrants concerning the QOW to the AM lab discussion period on **Tuesday, October 22**.

B. Objectives:

1. To gain experience with obtaining infrared spectra of solid compounds.
2. To gain experience with using solubilities of solid compounds in aqueous acid & base solutions in distinguishing among different compounds.
3. To discover a logical approach for determining if two solid samples contain the same compound.
4. To gain experience with interpreting IR spectroscopy to gain insight into the structure of an unknown compound.

C. Introduction:

In Experiment 5 you purified a sample of a solid organic compound by recrystallization so that you could identify the structure of the compound. However, there are fewer different compounds than there are students in each lab section. In fact, there is more than one student per compound and more than one compound per lab section. The ultimate goal of Experiment 6 is for each student to identify the group of labmates who have her/his compound. You have a pure sample that can be compared to the other samples in your lab section to determine which other lab mate(s) has (have) your compound. But what comparisons are likely to be useful in eliminating different compounds and identifying identical ones?

D. Question of the Week:

What measurements can allow us to distinguish one compound from another?

In formulating possible claims and warrants related to the QOW, consider the various measurement techniques we have used this semester, the introductory material on infrared spectroscopy and how a combination of these measurements might be used in this experiment to eliminate non-identical compounds and verify that compounds are identical.

Initial Claims and Warrants

1. Propose two analyses we have used this semester that might be useful in distinguishing between different compounds and/or confirming that two samples contain the same compound.
2. Provide your warrant for each proposal in 1.

E. Key Terms/concepts/techniques:

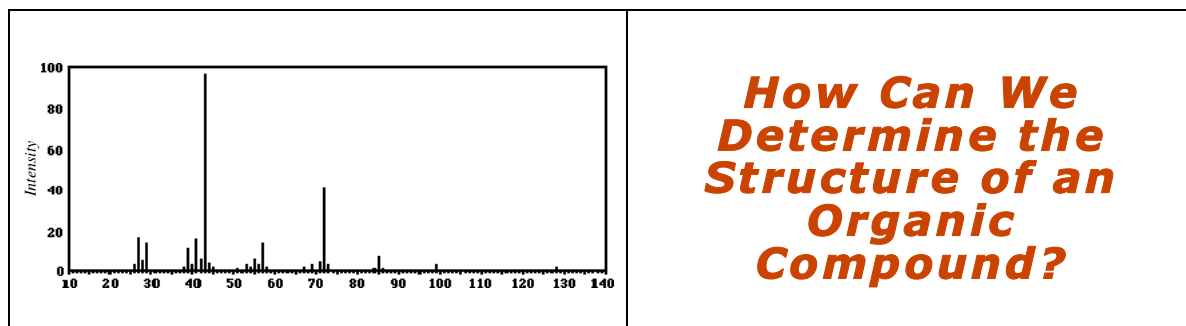
1. Infrared spectra.
2. Acid-Base solubility

F. Safety Matters:

1. Safety goggles are required.
2. Shoes that cover the tops of feet.
3. Clothing that covers from neck to knees.
4. Flammable liquids-no flames in the lab.

Experiment 7

Part A



**How Can We
Determine the
Structure of an
Organic
Compound?**

A. Pre-lab Preparation Assignment:

1. Read: Experiment 8 Part A, pp. 37-38.
2. Read: Lab Manual Part V. Appendix D1.
Padías pp. 106-107
CGW pp. 46-47
3. Read the Mass Spectrometry Background Introduction to Mass Spectrometry links on the Experiment 8A page of the course website.
4. Complete the electronic Prelab Questions (course website) by **10:00 PM, Monday, October 30** on the **Course Blackboard Site** as explained on the Course Information page.
5. Consider possible claims and warrants for the QOW (See below)
6. In your lab notebook: (See Lab Manual pp. 14-16 for format.)
 - a. Enter the title of the experiment, name of group members, the Objectives of the experiment and QOW on the appropriate first left-hand page.
 - b. Enter your initial claims and warrants concerning the QOW.
 - c. Create a Pre-Lab Notes section.
 - d. Up-date the Table of Contents.
7. Bring your notebook and your initial claims and warrants concerning the QOW to the AM lab discussion period on **Tuesday, October 31**.

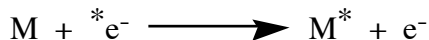
B. Objectives:

1. To gain experience with collecting mass spectra of solid compounds.
2. To gain expertise with analyzing IR and mass spectral data to determine the structure of organic compounds.

C. Introduction to Mass Spectrometry and Structural Analysis

1. Mass Spectral Analysis.

In Mass Spectral Analysis, organic molecules are bombarded by high energy electrons. Impact of an electron on a molecule transfers a large amount of energy to the molecule.



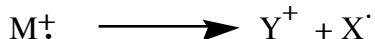
Equation 1: Electron Impact

One way that the molecule can dissipate the excess energy is to lose an electron and become a **Radical Cation** the "Molecular Ion".



Equation 2: Ionization

The mass spectrometer is designed to separate and count cations according to their **charge/mass ratio (m/z)**. Since most ions have lost only one electron, m/z = mass of the ion. So the mass of this first formed radical cation is equal to that of the original molecule entering the mass spectrometer and is referred to as the **molecular ion**.



Equation 3: Fragmentation

The molecular ion, because it still retains considerable energy from the initial collision, will tend to break down (fragment) into smaller fragments. These fragmentations usually result from breakage of one bond dividing the original structure into two pieces; one has a positive charge (a cation – Y^+) and the other contains the unpaired electron (a free radical – $X^\cdot$). The mass spectrometer detects only the positive ions and not the free radicals.

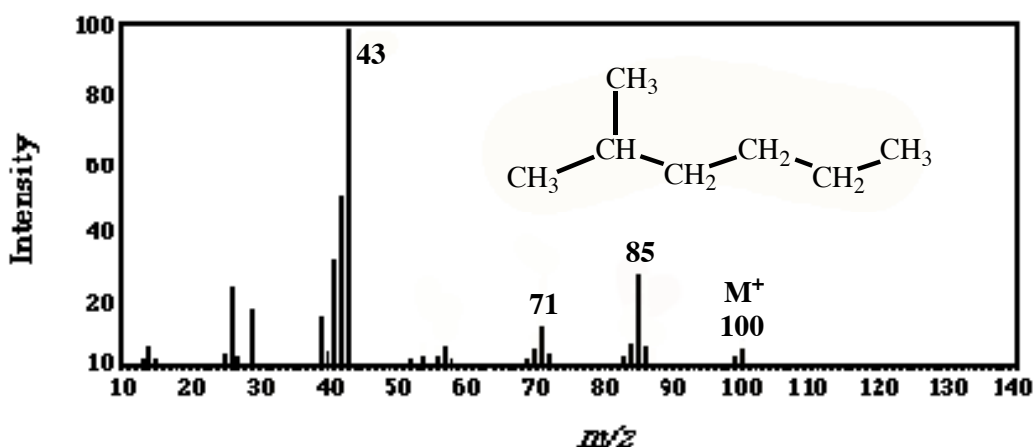


Figure 1: An Example of a Mass Spectrum of Pure compounds:

D. Question of the Day:

How do organic compounds interact with high energy electrons and what structural information can we obtain from the results?

After completing the pre-lab reading, consider how the effects of high energy electrons and analysis of the resulting molecular particles might assist in determining the structure of an organic molecule. In your analysis, consider what information is available in a mass spectrum.

Initial Claims and Warrants

1. Propose 2 characteristics of an unknown compound that might be determined from its mass spectrum.
2. Provide your warrant for each proposal in 1.

E. Key Terms/concepts/techniques:

1. Mass spectrometry
2. Base Peak
3. Molecular ion

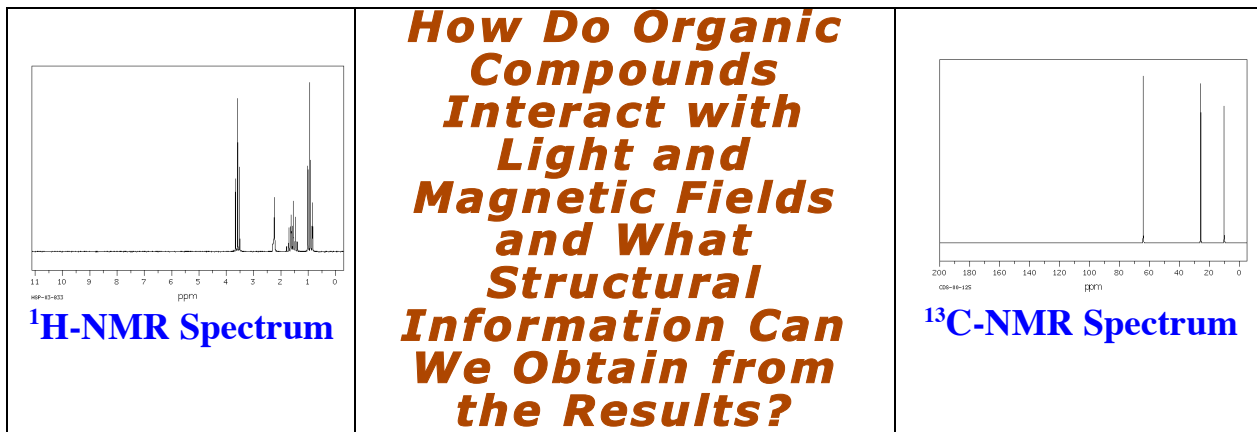
F. Safety Matters:

1. Safety goggles are required.
2. Shoes that cover the tops of feet.
3. Clothing that covers from neck to knees.
4. Flammable liquids-no flames in the lab.

Experiment 7

Part B

Week 1



A. Pre-lab Preparation Assignment:

1. Read: Experiment 7 Part B Week 1, pp. 39-40.
2. Read: Lab Manual Part V. Appendix E1-E2.
3. Read: Introduction to NMR spectroscopy link developed by Prof. William H. Reusch of Michigan State University (Link on experiment 7 Part B page of the course website.), The Background and Chemical shift sections up to “For the properties of some common NMR solvents [Click Here](#).”
4. Complete electronic Prelab-1 Questions (course website) by **10:00 PM, Monday, November 4** on the **Course Blackboard Site** as explained on the Course Information page.
6. Consider possible claims and warrants for the QOW (See below)
7. In your lab notebook: (See Lab Manual pp. 14-16 for format.)
 - a. Enter the title of the experiment, name of your group, names of group members, the Objectives of the experiment and QOW on the appropriate first left-hand page.
 - b. Enter your initial claims and warrants concerning the QOW.
 - c. Create a Pre-Lab Notes section.
 - d. Up-date the Table of Contents.
8. Bring your notebook and your initial claims and warrants concerning the QOW to the AM lab discussion period on **Tuesday, November 5**. Also bring your 7A In-Lab questions and personal paper unknown sheets.

B. Objectives:

1. To gain experience with collecting NMR spectra.
2. To gain experience with analyzing NMR spectral data to determine the structure of organic compounds.

C. Introduction:

NMR will be the third spectroscopic method you have explored to see how it may assist in identifying unknown compounds.

D. Question of the Week:

How do organic compounds interact with light and magnetic fields and what structural information can we obtain from the results?

In preparing claims and warrants to address the QOW, consider the different aspects of structure probed by IR, MS and NMR.

Initial Claims and Warrants

1. Propose 2 specific new characteristics of an unknown compound that might be determined from NMR that cannot be determined from its IR and MS spectrum.
2. Provide your warrant for each proposal in 1.

D. Key Terms/concepts/techniques:

- Nuclear Magnetic Resonance Spectroscopy
- Chemical Shift
- Signal Integration

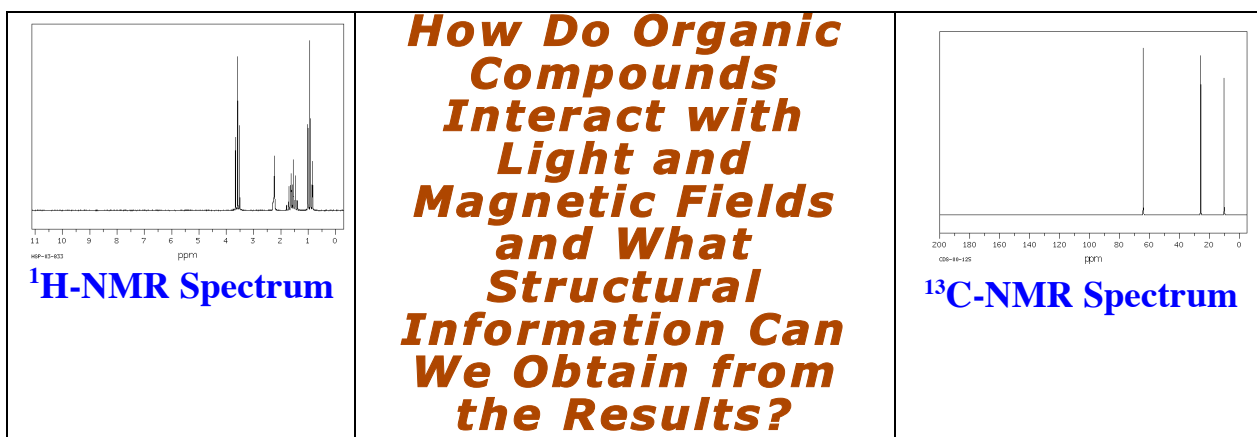
F. Safety Matters:

- a. Safety goggles are required.
- b. Shoes that cover the tops of feet.
- c. Clothing that covers from neck to knees.
- d. Flammable liquids-no flames in the lab.

Experiment 7

Part B

Week 2



B. Pre-lab Preparation Assignment:

1. Read: Experiment 7 Part B Week 2, p. 41-43.
2. Work Through: Lab Manual Part V. Appendix E5-E9 to explore the data in Table 2 E7-E8.
4. Complete electronic Prelab-2 Questions (course website) by **10:00 PM, Monday, November 11** on the **Course Blackboard Site** as explained on the Course Information page.
5. Consider possible claims and warrants for the QOW (See below)
6. In your lab notebook: (See Lab Manual pp. 14-16 for format.)
 - a. Enter the title of the experiment, name of group, the Objectives for the experiment and QOW on the appropriate first left-hand page.
 - b. Enter your initial claims and warrants concerning the QOW.
 - c. Create a Pre-Lab Notes section.
 - d. Up-date the Table of Contents.
7. Bring your notebook and your initial claims and warrants concerning the QOW to the AM lab discussion period on Tuesday, November 16. Be sure to also bring your lab manual.

B. Objective:

To gain experience with analyzing NMR spectral data to determine the structure of organic compounds.

C. Introduction:

Last week we developed a sense of the types of information that can be obtained from NMR observations of proton and carbon atoms in organic compounds. NMR provides a very rich data set on most compounds and it is easy to get confused as to where to start in analyzing the data.

D. Question of the Week:

What structural information can be obtained from NMR spectra without chemical shift assignments?

This week's QOW suggests that you think about what can be gleaned from an initial analysis of an NMR spectrum without considering the specific chemical shift values of the signals.

Initial Claims and Warrants

1. Propose three structural characteristics of an unknown molecule that might be determined from its NMR spectrum without considering the specific chemical shift values of the peaks.
2. Provide your warrant for each proposal in 1.

D. Key Terms/concepts/techniques:

- Nuclear Magnetic Resonance Spectroscopy
- Number of Absorptions
- Signal Integration
- Multiplicity of ^1H -NMR signals (spin-spin splitting)

F. Safety Matters:

- a. Safety goggles are required.
- b. Shoes that cover the tops of feet.
- c. Clothing that covers from neck to knees.
- d. Flammable liquids-no flames in the lab.

Part V. Techniques and Theory

Appendix A.

Classification of unknown compounds from their solubilities in aqueous media:

Observations:

1. Solubility in Water

Most organic compounds are insoluble in water, however, polar compounds that have no more than about 5 carbon atoms for every oxygen, nitrogen or sulfur atom are usually at least slightly soluble in water. If the compound does dissolve, the pH of the resulting solution will suggest whether the compound is strongly acidic ($\text{pH} < 4$), strongly basic ($\text{pH} > 9$) or essentially neutral ($\text{pH} \geq 4$ and ≤ 9).

2. Solubility in Base (1M NaOH)

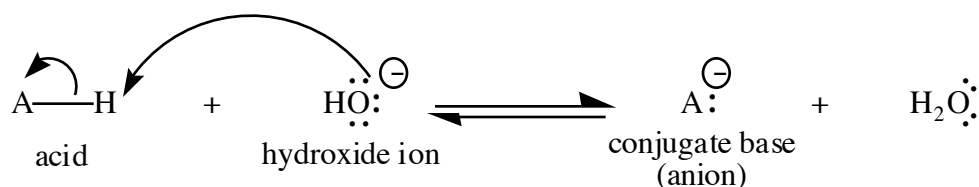
Acidic compounds that are insoluble in water are generally more soluble in 1M NaOH than they are in water.

3. Solubility in Acid (1M HCl)

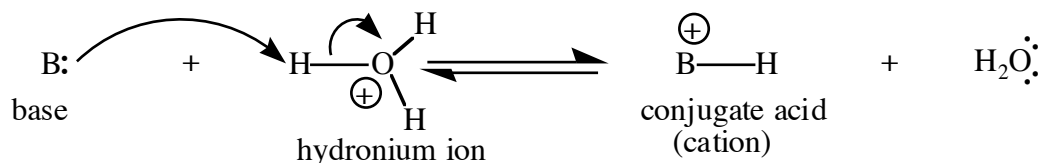
Basic compounds that are insoluble in water are generally more soluble in 1M HCl than they are in water.

Warrants & Backing:

Basic Solutions: Acidic compounds react with hydroxide ion losing a proton and becoming negatively charged ions (anions).



Acidic Solutions: Basic compounds react with the hydronium ion, which is produced by HCl in water, gaining a proton and becoming positively charged ions (cations).



Theory: In the process of dissolving a solute, water must create “holes” in its H-bonded structure. Creation of the holes disrupts some of water’s strong hydrogen bonds and reduces the randomness of the remaining ones. Thus, dissolving solutes will raise the free energy ($\Delta G > 0$) of water ($\Delta H > 0$ & $\Delta S < 0$). If the solute molecules can replace some of the lost water hydrogen bonds with solute-solvent hydrogen bonds or ion-dipole interactions, water’s ΔG increase is moderated making solubility possible.

Since water interacts very favorably with both positive and negative ions (ion-dipole interactions), charged species are more soluble in water than are similar structures with no charge. Thus, increased solubility of acidic compounds in basic solutions and basic compounds in acidic solution result from water’s favorable interactions of the ionic forms of these compounds produced by the above acid-base reactions.

Appendix B.

Extraction (See also, Padías pp. 128-131)

Sample Acid-Base Extraction Procedure for Separation of a Mixture containing an Acid, a Base and a Neutral Compound

This procedure is designed to separate a ternary mixture of organic compounds using acid and base extractions. This example is provided to give you some guidance for both acidic and basic extraction procedures.

Procedure:

Be sure to record all pH measurements and all weights of isolated products in your Results Section. Be sure compounds are dry before weighing.

1. Dissolution of Sample.

Weigh out, to the nearest thousandth of a gram, approximately 2 g of the mixture. Dissolve the mixture in ~50 mL of dichloromethane (CH_2Cl_2). If any solid doesn't dissolve, add an additional 25 mL of CH_2Cl_2 . If undissolved solid still remains, add the first extracting solvent (1.0 M HCl or 1.0 M NaOH) and swirl the mixture. Finally filter any remaining solid (by gravity) allowing the filtrate to flow directly into a separatory funnel. Otherwise, pour the solution into the separatory funnel. Keep the recovered undissolved solid for possible further analysis. (One component of the mixture may be insoluble in dichloromethane.)

2. Acid Extraction.

Extract the CH_2Cl_2 solution two times with 25 mL aliquots of 1.0 M HCl. Test the pH of the aqueous solution from the second extraction. It should be as acidic as the original 1.0 M HCl solution. If it is not, extract the CH_2Cl_2 solution again with 25 mL of 1.0 M HCl. Combine the aqueous layers from all HCl extractions in a 250 mL beaker and label the beaker "acid extracts".

3. Basic Extraction.

Extract the CH_2Cl_2 solution resulting from step 2, two times with 25 mL aliquots of 1.0 M NaOH. Test the pH of the aqueous solution from the second extraction. It should be as basic as the original 1.0 M NaOH solution. If it is not, extract the CH_2Cl_2 solution again with 25 mL of 1.0 M NaOH. Combine the aqueous layers from all NaOH extractions in a 250 mL beaker and label the beaker "base extracts".

4. Recovery of the Compounds.

a. CH_2Cl_2 solution:

Dry the CH_2Cl_2 solution resulting from step (3.) with anhydrous Na_2SO_4 . Let this mixture stand for about 5 min., swirling it frequently. Remove the drying agent by decanting the solution into a clean dry 125 mL Erlenmeyer flask, add a boiling stone and evaporate the solvent in a hot water bath in a hood. Transfer the residue to a vial and allow it to dry. (See section d. below)

b. Acid Extracts:

While the CH_2Cl_2 is evaporating, cool the acidic solution from step (2.) in an ice bath to help dissipate heat that will be generated in the neutralization process. Make the solution basic by slowly adding 6 N NaOH until the solution tests strongly basic with pH paper. A precipitate should form during the addition of NaOH. Collect the solid by vacuum filtration and wash the solid on the filter with cold water. Transfer the compound to a watch glass and allow it to dry. (See section d. below)

c. Base Extracts:

Cool the basic solution from step (3.) in an ice bath to help dissipate heat that will be generated in the neutralization process. Make the solution acidic by slowly adding 6 M HCl and stirring until it tests strongly acidic with pH paper. Collect the precipitated solid by vacuum filtration and wash the solid on the filter with cold water. Transfer the compound to a watch glass and allow it to dry. (See section d. below)

d. Drying Procedures:

The compound recovered from the CH_2Cl_2 solution should be crushed to small particle size and allowed to dry for a total of about 1 hour. It will dry quickly since it was isolated from a low-boiling solvent.

The compounds recovered from the acid and base extracts should be allowed to air dry in your desk until the next period. These compounds were isolated from water and water evaporates slowly from these polar compounds.

5. Sample Analysis and Submission:

Determine the mass of the compound recovered from the CH_2Cl_2 solution. Record your data in your Results Section, place your sample in an appropriately labeled vial (See Lab Manual Part II, p. 12), and seal it tightly. Submit your sample according to your instructor's directions.

Next Lab Period:

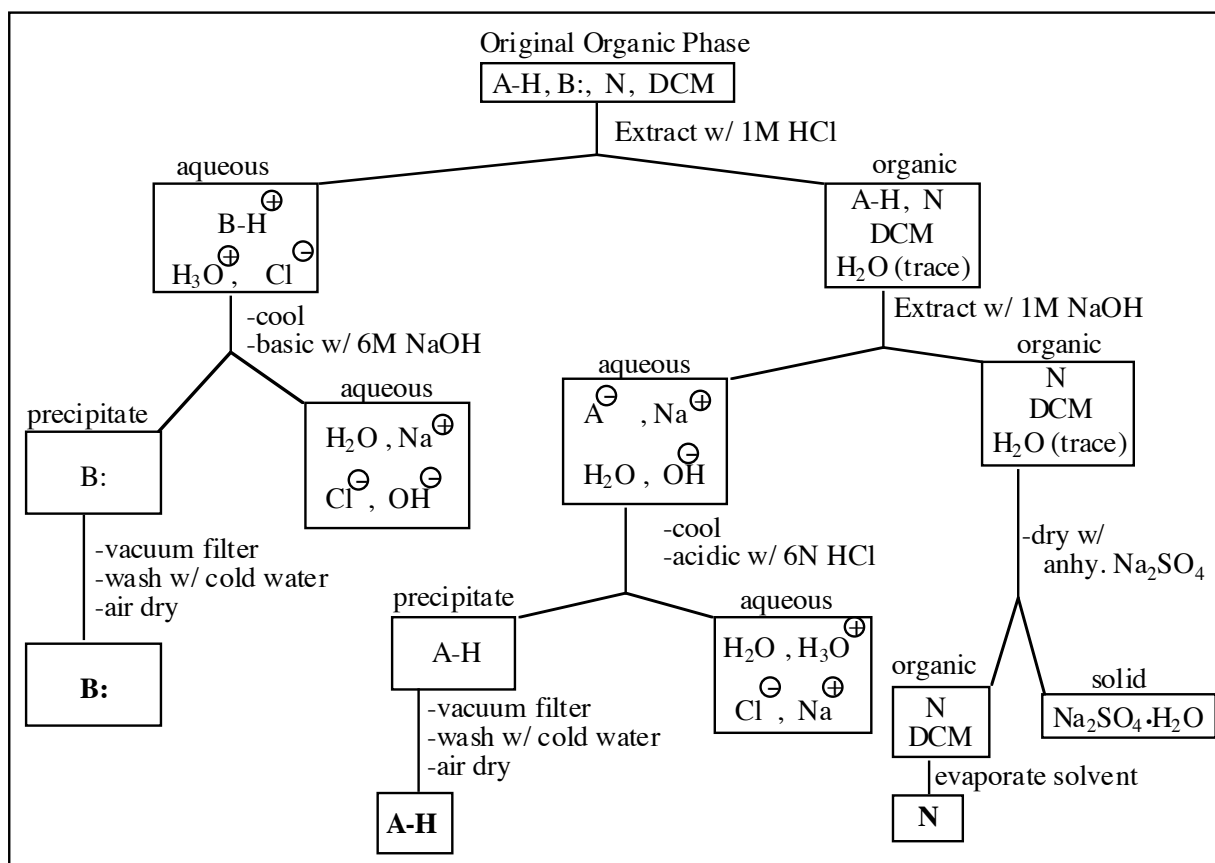
After the compounds you recovered from the aqueous solutions have had sufficient time to dry completely, determine their masses, place each in a labeled vial, and submit the samples as with that recovered from CH_2Cl_2 .

Sample Flow Diagram

Flow Diagram for Separation of the Mixture of an Acid, a Base and a Neutral Compound described in the procedure on B-1 & B-2. (See also, Padías pp. 120 - 121)

Abbreviations

A-H = acid	N = neutral compound
B: = base	DCM = dichloromethane



Appendix C. Infrared Spectroscopy (IR Spectroscopy):

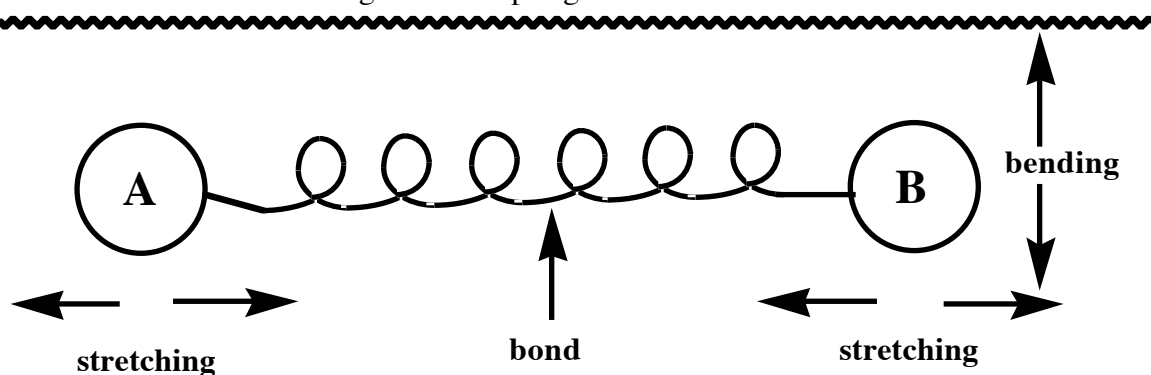
See also See Padías pp. 66 – 74 and CGW pp. 63-72

Spectroscopy is the study of the interaction of matter with electromagnetic radiation (light). We identify different types of spectroscopy by the region of the electromagnetic spectrum they use to probe matter. IR spectroscopy use light in the infrared region ($4000 - 400 \text{ cm}^{-1}$)

Source of the IR Spectrum

Vibrational Energy Levels

Covalent bonds link atoms together to form molecules. Though these bonds have normal average lengths, the relative positions of the atoms are constantly changing due to bond vibrations. A bond can be thought of as a spring with atoms attached to each end.

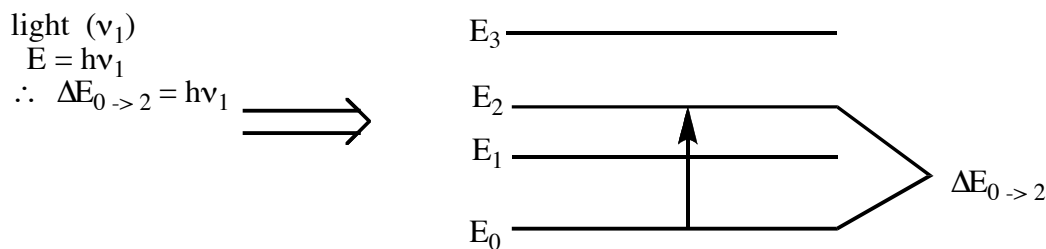


As with other types of molecular changes, bond vibrations occur only at certain frequencies (ν) and each of these ν 's is associated with a vibrational energy level of energy = $h\nu$ (h = Planck's constant). Thus, each bond has a series of vibrational energy levels.

Transitions

When electromagnetic radiation (light) is shone upon a molecule, one of its bonds can absorb a quantum of the energy and pass from a lower vibrational energy level to a higher one if the radiation contains light of a proper frequency (ν). Such energy level transitions are the fundamental bases for all types of spectroscopy.

e.g.



The Spectrum:

IR absorption bands are measured as a function of the ν of the incident light. The units of ν are cm^{-1} . (See Figure 6 on p. 17 of this lab manual)

Note: Since $E = h\nu$ for light, the energy of absorbed light is proportional to ν , that is, the energy of the photons of light increase as their ν 's increase.

Assignment of IR Vibrational ν 's to Specific Bonds in Molecules:*Data:*

Symbols used in Figure 1:

st = strong (high intensity) absorption	wk = weak intensity absorption
sh = sharp (narrow peak)	br = broad (wide peak)

Figure 1: Vibrational Frequencies of some Organic Molecules

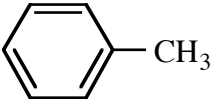
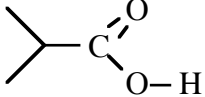
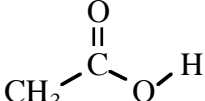
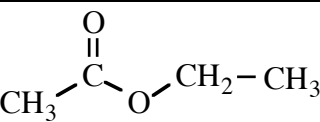
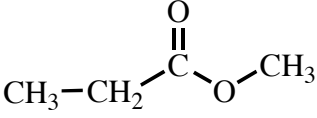
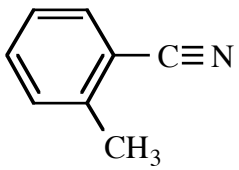
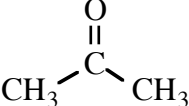
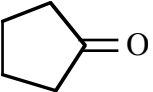
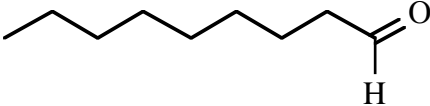
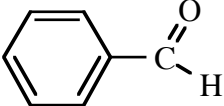
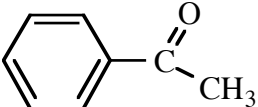
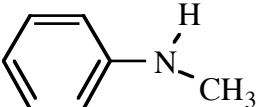
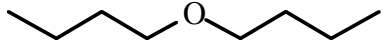
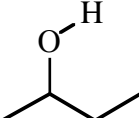
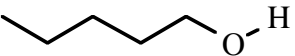
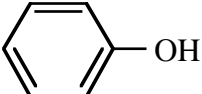
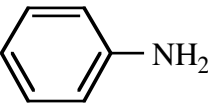
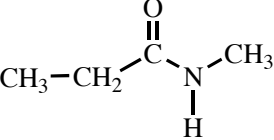
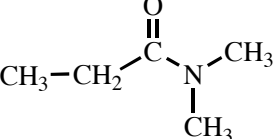
	STRUCTURE	Important IR ν 's (cm^{-1})
1.	$\text{CH}_3(\text{CH}_2)_6\text{CH}_3$	2945
2.	$(\text{CH}_3)_2\text{C}=\text{C}(\text{CH}_3)(\text{CH}_2)_6\text{CH}_3$	2950, 1645 wk
3.	$\text{CH}_2=\text{C}(\text{CH}_3)_2$	3075, 2935, 1660 wk
4.	$\text{CH}_3(\text{CH}_2)_3\text{C}\equiv\text{C}-\text{H}$	3268 sh , 2945, 2110 (sh & wk)
5.	$\text{CH}_3(\text{CH}_2)_2\text{C}\equiv\text{C}-\text{CH}_3$	2950, 2200 (sh & wk)
6.		3040, 2925, 1600, 1500, 1460
7.		3030, 2940, 1510, 1450
8.		3300 $\rightarrow$ 2900 (br & st), 2955, 1710 st , 1240
9.		3300-2500 (br & st), 2960, 1710 st , 1290
10.		2970, 1740 st , 1230, 1100
11.		2945, 1740 st , 1250, 1100
12.	$\text{CH}_3(\text{CH}_2)_3\text{C}\equiv\text{N}$	2945, 2250 sh
13.		3035, 2925, 2210 sh , 1600, 1490, 1460
14.		2930, 1725 st

Figure 1 (Continued)

	STRUCTURE	Important IR ν 's (cm^{-1})
15.		2920, 1740 st
16.		2960, 2850 sh , 2700 sh , 1720 st
17.		3055, 2800 sh , 2750 sh , 1700 st , 1600, 1580, 1450
18.		3045, 2950, 1680 st , 1600, 1580, 1450, 1425
19.	$\text{CH}_3(\text{CH}_2)_3\text{-NH}_2$	3360, 2945, 1160
20.	$(\text{CH}_3\text{CH}_2\text{CH}_2)_3\text{N}$	2955, 1180, 1080
21.	$(\text{CH}_3\text{CH}_2\text{CH}_2)_2\text{NH}$	3300, 2940, 1130
22.		3400, 3030, 2960, 1600, 1500, 1480, 1080
23.		2945, 1120
24.		3400-3200 (br & st), 2950, 1100
25.		3450-3250 (br & st), 2940, 1060
26.		3600-3000 (br & st), 3050, 1600, 1580, 1500, 1480, 1250
27.		3400, 3350, 3050, 1610, 1590, 1495, 1480, 1180
28.		3225, 2955, 1685 st , 1150, 1110
29.		2960, 1690 st , 1200, 1130

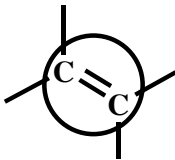
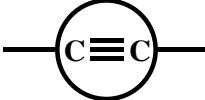
Exploration

1. Recognizing that Infrared light absorption results in changes in vibrational states of specific bonds in molecules, we should be able to relate many of the IR absorption frequencies to the bonds present in the absorbing molecules. The most common atoms in organic molecules are C, H, O & N. On the next two pages is a list of the commonly observed types of bonds found in organic molecules. Follow the suggested steps below to compare the IR absorption frequencies found in the 29 compounds in Figure 1 and assign approximate absorption frequencies ($\pm 10 \text{ cm}^{-1}$) to each of the types of bonds. (circled bond in each structure) For each assignment, provide references to the #'s of the compounds from Figure 1 that you used to make the assignment and provide your warrant. (Note that possible substituents on bonds with no atoms explicitly indicated in the list below could be C or H)

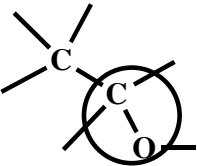
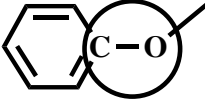
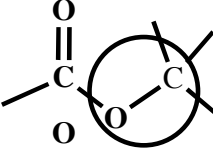
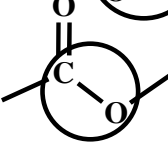
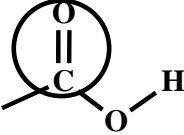
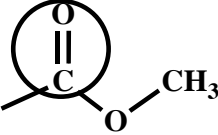
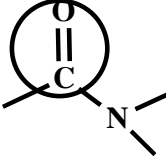
There are many ν 's in Figure 1. In beginning to assign these frequencies, consider:

- a. Which compound is the simplest place to start? Provide your warrant.
- b. How does that compound limit possible assignments? Provide your warrant.
- c. Which compounds appear to be best to consider next? Provide your warrant.
- d. What assignments are possible by combining ν 's from these compounds with the one from a? On pp. C5 & C6, add the ν 's of the bonds that you can assign and provide your warrant.
- e. Where can you go from there? Provide your warrant.
- f. Continue the process to assign ν 's to all bonds designated on pp. C5 & C6.

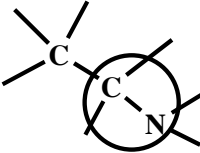
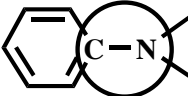
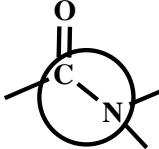
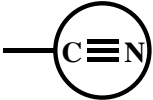
Carbon-Carbon Bonds

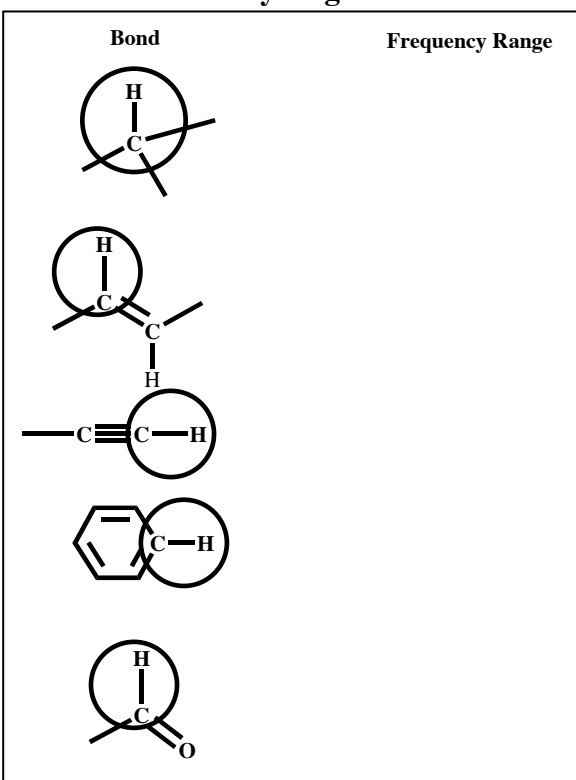
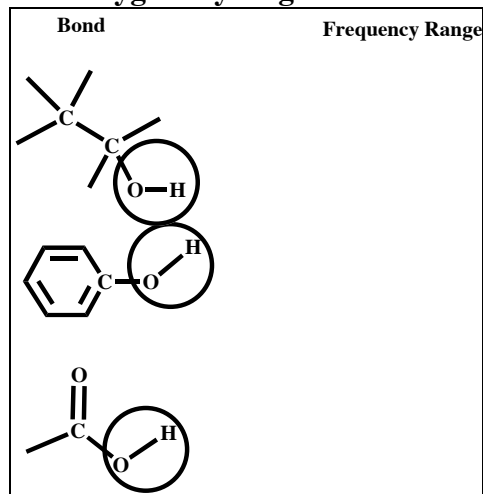
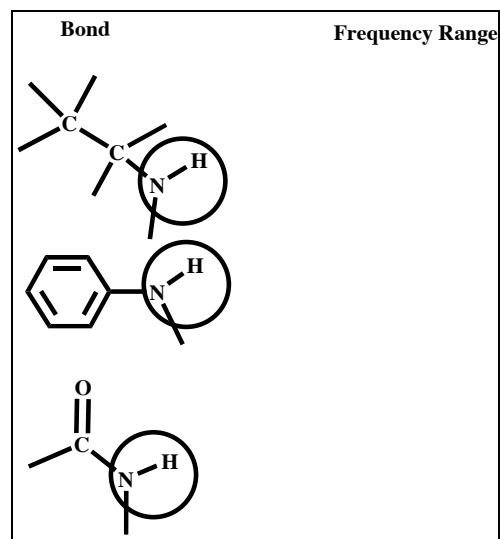
Bond	Frequency Range
	
	
	

Carbon-Oxygen Bonds

Bond	Frequency Range
	
	
	
	
	
	
	
	

Carbon-Nitrogen Bonds

Bond	Frequency Range
	
	
	
	

Carbon-Hydrogen Bonds**Oxygen-Hydrogen Bonds****Nitrogen-Hydrogen Bonds**

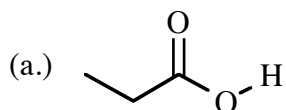
2. Padías groups molecular IR absorption frequencies according to functional groups present. (Table 2-1 p. 72) Although this arrangement can be helpful in identifying peaks expected from the presence of a functional group, often we have a spectrum, like those in Padías Figures 2-22 – 2-25 (pp. 75-76), of an unknown compound and need to use absorptions frequencies to identify likely functional groups present. So it is also useful to divide the IR frequency region into sub-regions and look at the types of bonds that absorb in each sub-region.

Use your frequency assignments in 1. to assign the bond types to each of the sub-regions listed below. Not that specific bond (e.g. C-H) absorb over a range of ν 's. Note these ranges with the bond types.

Region	Bond types
1: (3600-3200 cm^{-1})	
2: (3100-2500 cm^{-1})	
3: (2500-2000 cm^{-1})	
4: (1750-1630 cm^{-1})	
5: (1350-1000 cm^{-1})	

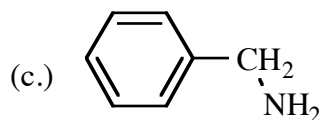
Applications:

1. As you noticed in your above analyses, many organic functional groups contain more than one type of bond. It is useful to recognize the pattern of IR absorptions that are related to each functional group. For the following structures, identify the important functional groups and use your assignments from the *Exploration* above to predict approximate values of IR ν 's characteristic of the functional groups and provide your warrant for the assignments:



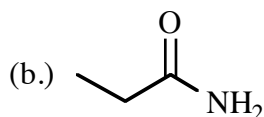
Functional group(s):
Bond Types:
Expected ν 's (cm^{-1}):

Warrant:



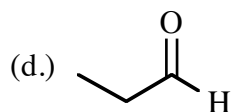
Functional group(s):
Bond Types:
Expected ν 's (cm^{-1}):

Warrant:



Functional group(s):
Bond Types:
Expected ν 's (cm^{-1}):

Warrant:



Functional group(s):
Bond Types:
Expected ν 's (cm^{-1}):

Warrant:

2. What functional groups are likely to be present in each of the following compounds? Provide your warrant to support your predictions.

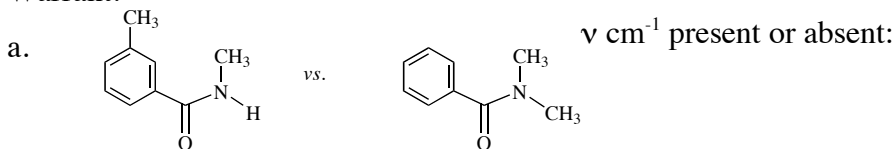
a. $\text{C}_7\text{H}_6\text{O}$ 3050, 2820, 2740, 1705 (**st**), 1600, 1580, 1430 cm^{-1}
Bond Types Present:
Likely Functional group(s):
Warrant:

b. $\text{C}_{11}\text{H}_{12}\text{O}_2$ 3030, 2960, 1700 (**st**), 1630, 1560, 1480, 1440, 1160 cm^{-1}
Bond Types Present:
Likely Functional group(s):
Warrant:

c. $\text{C}_5\text{H}_8\text{O}$ 2935, 1670 **st**, 1620 cm^{-1}
Bond Types Present:
Likely Functional group(s):
Warrant:

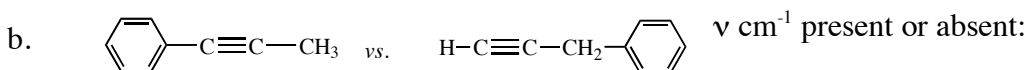
d. $\text{C}_{10}\text{H}_{14}$ 3030, 2940, 1520, 1460, 1420 cm^{-1}
Bond Types Present:
Likely Functional group(s):
Warrant:

3. Because IR spectra detect specific bond vibrations, it is a useful technique for differentiating between similar structures that differ only in the presence of one bond type. This application of IR spectra is illustrated in this question. For each of the following pairs of structures, if you had the IR spectrum that had to be from one of the compounds, how could you use the spectrum to distinguish between the two possible structures in each pair? Provide your Warrant.



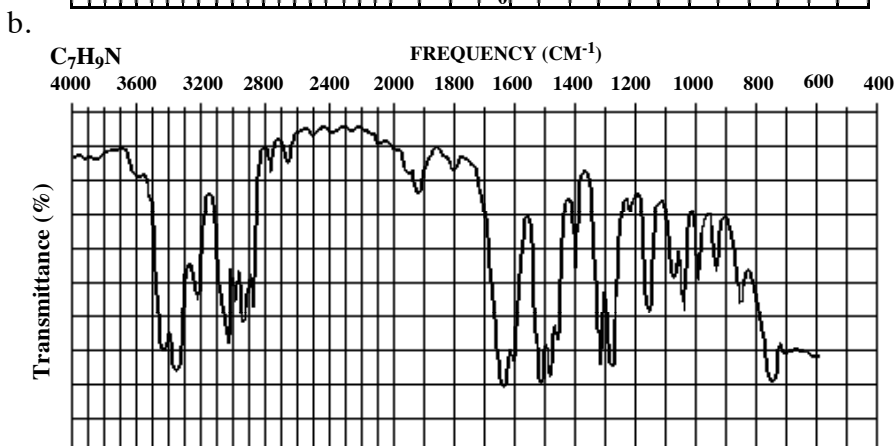
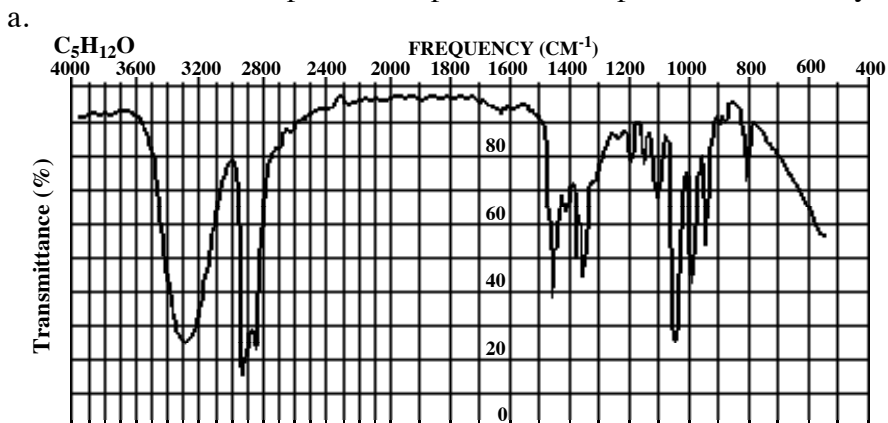
Warrant:

ν



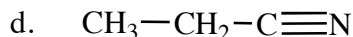
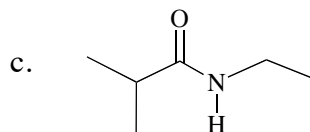
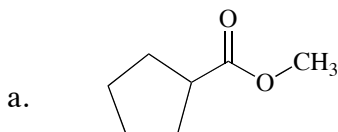
Warrant:

4. So far you have worked with IR data presented as numerical frequencies. In the lab you will collect IR spectra in a graphical representation. The graphical presentation requires that you interpret the spectrum to translate the graph into numerical designations for peaks to assist in your analysis. This question gives you some experience with interpreting graphical IR spectra. For each of the following spectra, assign ν 's to the major peaks and then suggest functional groups that might be present. Finally give one or two possible molecular structures for the compound that produced the spectrum. Provide your Warrant.



IR Additional Out of Class Applications:

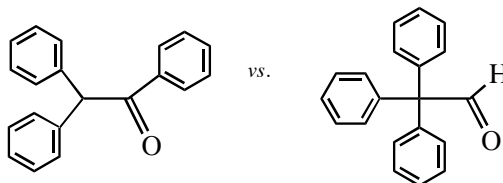
1. Predict approximate values of important IR ν 's for the following compounds:



2. What functional groups are likely to be present in each of the following compounds? Explain your logic.

(1.)	$\text{C}_4\text{H}_6\text{O}$	2940, 2800, 2720, 1680 (st), 1630 cm^{-1}
(2.)	$\text{C}_7\text{H}_8\text{O}$	3300 (br & st), 3040, 2940, 1610, 1570, 1480, 1460, 1160 cm^{-1}
(3.)	$\text{C}_9\text{H}_{10}\text{O}$	3020, 2955, 1690 (st), 1600, 1590, 1460 cm^{-1}
(4.)	$\text{C}_7\text{H}_7\text{NO}_2$	3460, 3360, 3000 (br-st), 1670, 2950, 1640, 1180 cm^{-1}

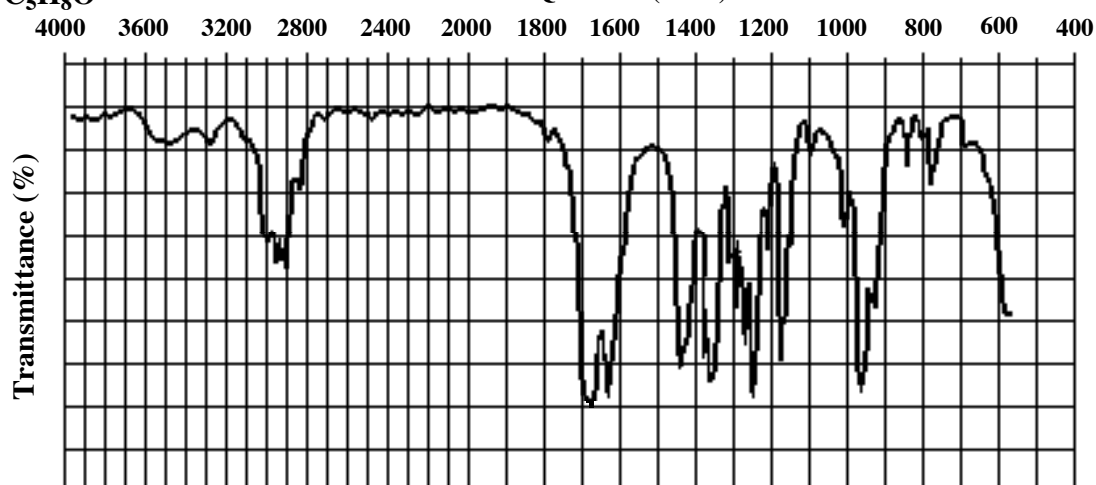
3. For the following pair of structures, if you had the IR spectrum of a reaction product that had to be one of the compounds, how could you use the spectrum to distinguish between the two possible structures? Explain how.



4. What functional groups might be present in the molecule that produced the following set of data? Give one or two possible structures that are consistent with the molecular formula and the spectrum. Provide your warrant.

$\text{C}_5\text{H}_8\text{O}$

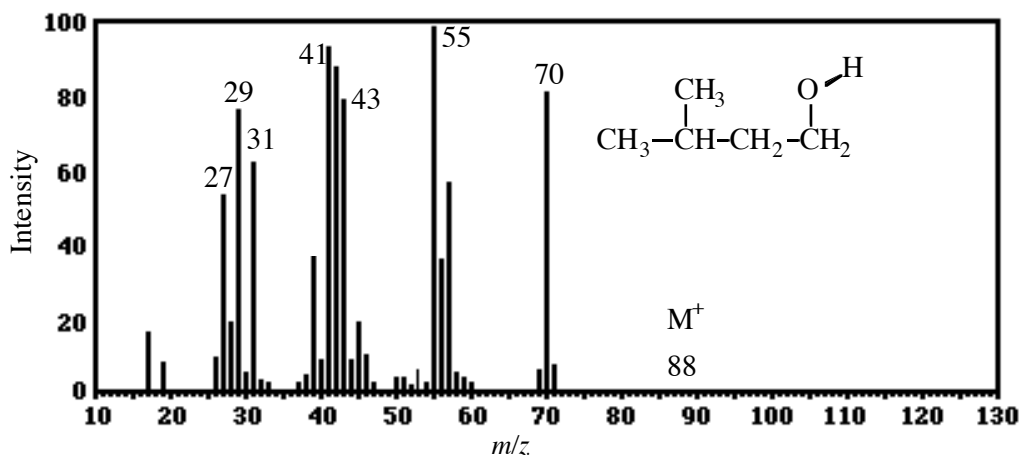
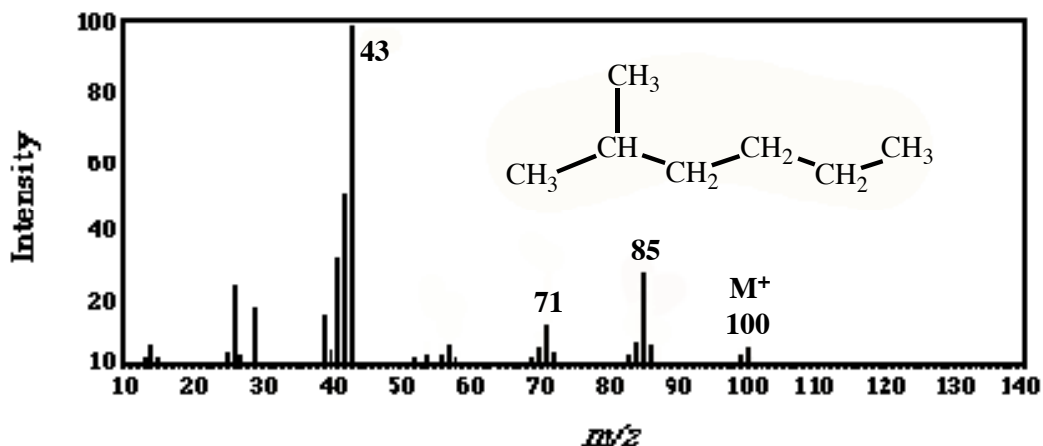
FREQUENCY (CM^{-1})



Appendix D. Mass Spectrometry

Mass Spectra Theory & Practice (See also: Padías pp. 106-113)

Examples of Mass Spectra of Pure compounds:



Analysis of Mass Spectra.

The Introduction to Mass Spectrometry presented in Experiment 7 Part A indicated that after being struck by high energy electrons, molecules tend to break up. So the mass peaks that are smaller than the molecular mass in the above spectra probably come from pieces of the original molecule. An analysis of these “fragmentation peaks” should provide information about the structure of the original molecule. This “fragmentation process” can be represented by the equation below. After the molecule has been struck by the high energy electron and lost an electron it has a single + charge and an unpaired electron. So if the molecule fragments, one of the resulting pieces will have the + charge (Y^+) and the other piece will have an unpaired electron, but no charge ($X\cdot$).

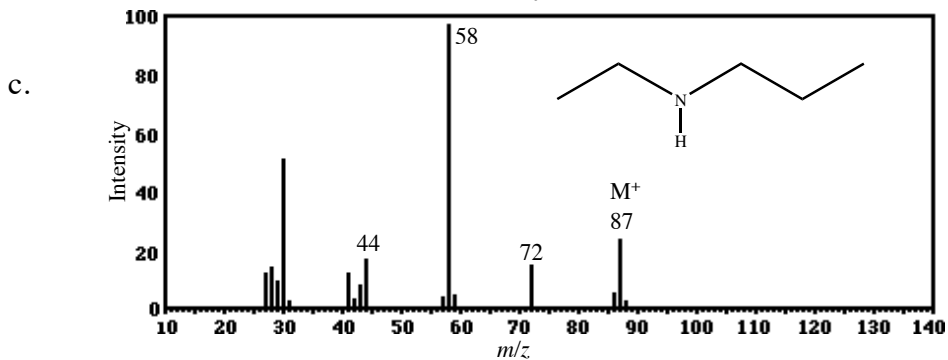
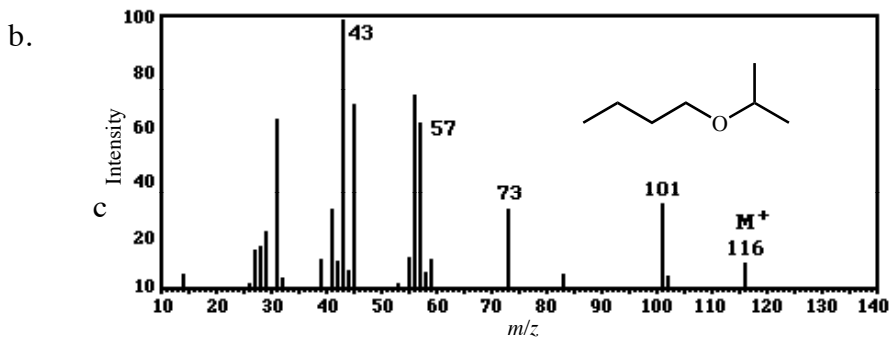
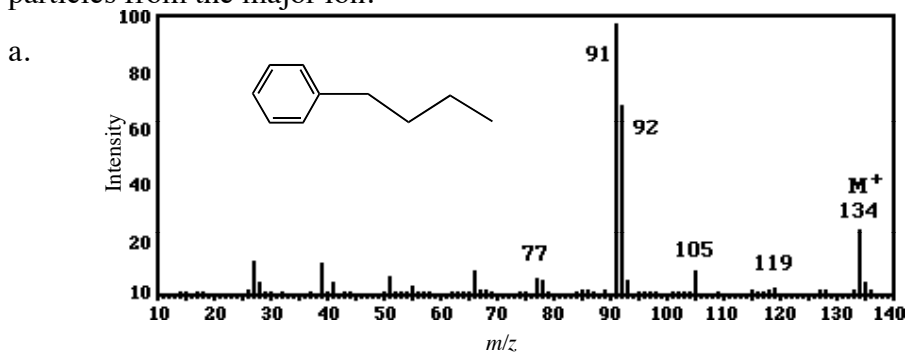


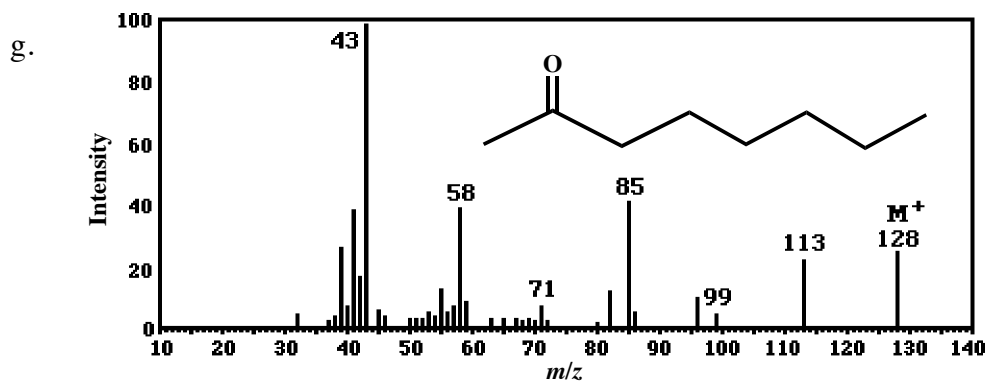
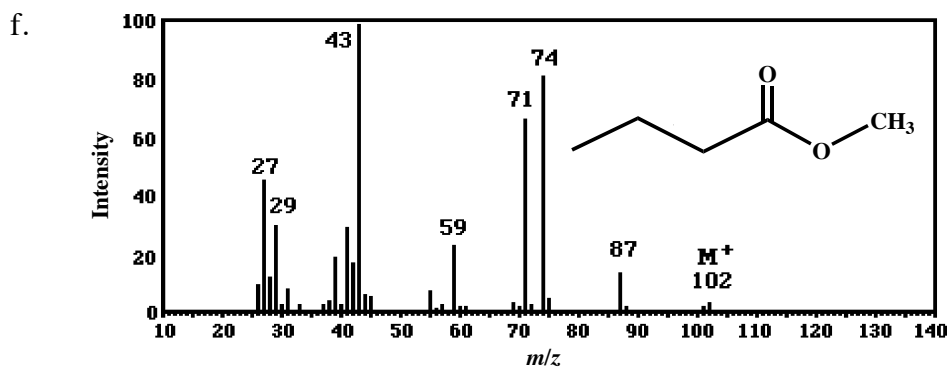
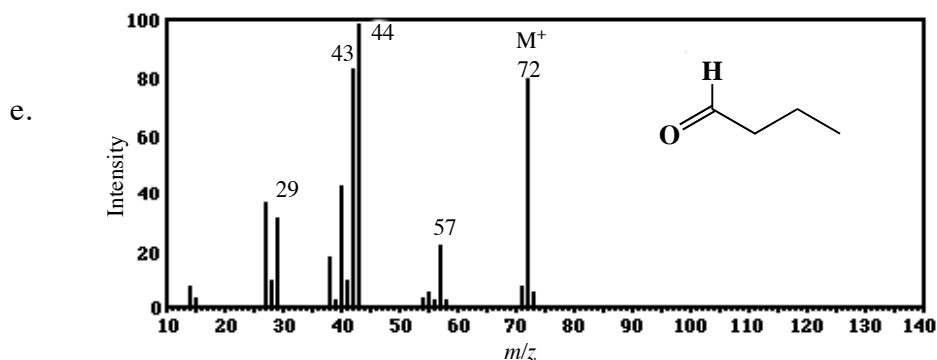
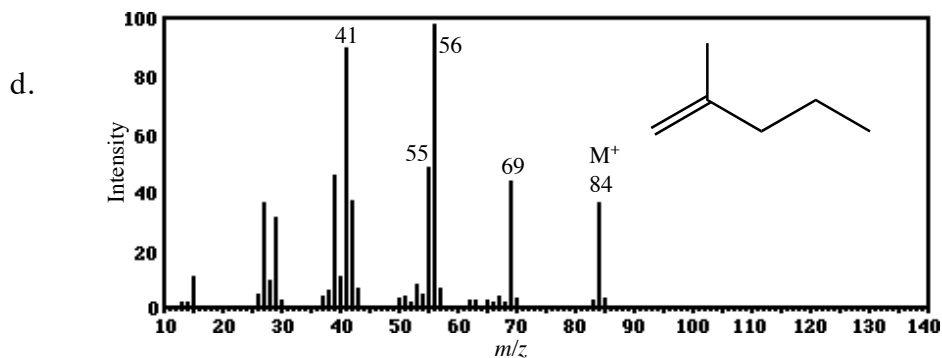
Mass spectrometers are designed to detect only positively charged particles. So, the mass of Y^+ can be determined from a peak in the mass spectrum of M , but the mass of $X\cdot$ is not detected by the mass spectrometer. However, the masses of $Y^+ + X\cdot =$ the mass of M^+ , so the mass of $X\cdot$ can be determined by subtracting the mass of the detected peak for Y^+ from the mass of the molecular ion (M^+). Thus, the peak at $m/z = 31$ in the spectrum above arises

from the splitting of the molecular ion into an + ion of mass 31 and a neutral radical of mass 57 ($M-31$). Often the radical is smaller than the ion and can be identified in the molecular structure more easily. **Table 2-8 on p. 111 in Padías** gives masses of many common neutral radicals and cations formed from molecular ion fragmentations.

Exploration of Mass Spectral Fragmentation Patterns.

1. Look carefully at each of the two spectra on D1 and the 7 additional spectra below and on pp. D3 $\rightarrow$ D4. Considering the structure of the compound, the brief introduction to mass spectrometry on pp. D1 & D2 and in Padías & CGW, **try to identify the structures of the ions** whose masses are indicated with a number in each mass spectrum. Assuming that the **ions** are produced by **breakage of only one bond** in the original structure, **identify one bond that must have broken to form each ion**. For large mass ion peaks, you **may need to use the $[M^+ - (\text{ion peak mass})]$ value** to identify the radical ($X^\bullet$ in equation 1) first. You will note that there are other peaks "clustered" around the designated peaks. These peaks often arise from loss of hydrogen atoms or other small particles from the major ion.

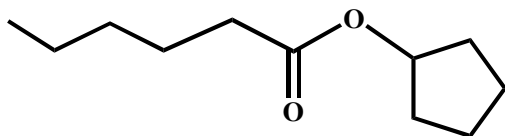
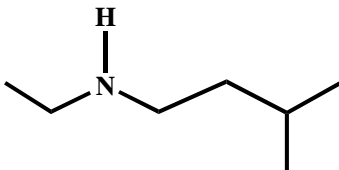
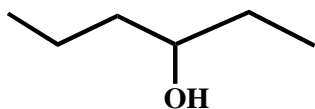
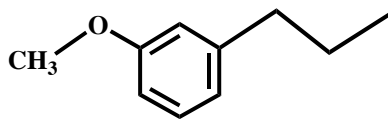
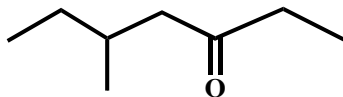




2. Review your bond breakage assignments in 1a.-g. and suggest some guidelines for predicting sites of breakage and ion masses in the mass spectra of any given compound. Compare your guidelines with those on pp. D-5 – D6

Application:

- a. Use your guidelines developed above to help you predict the masses of major peaks that you would expect in the mass spectrum of each of the following structures. Provide your warrant for each predicted peak.



- b. Use your experience in the *Exploration* to help you identify as many molecular fragments present in the molecules with the following molecular formulas and mass spectral peaks. Then suggest possible structures of molecule that could have produced the spectra. For each structure suggested, explain what data and logic led you to the prediction.

(1.)


$$m/e = 102, 87, 73, 59, \text{ \& } 43$$

(2.)


$$m/e = 73 \text{ \& } 30$$

Fragmentation Patterns**Guidelines for Predicting and Understanding the Most Prominent Peaks in the Mass Spectrum of a Compound:**

The following guidelines are provided to help you organize your discoveries in the *Exploration*. Read through them to see how they compare with your conclusions about Mass Spectral breakage sites in organic molecules. Neither Padías nor CGW deal with this approach to the analysis of Mass Spectra.

1. Guidelines for predicting molecular fragmentations:**a. Loss of Small Molecules or Radicals:**

- e.g. The mass spectrum of methyl butyl ether, below, shows a prominent peak at $m/z = 57$ due to loss of a neutral methoxy radical (mass of 31) from the molecular ion.

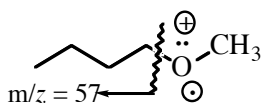
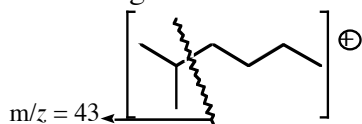


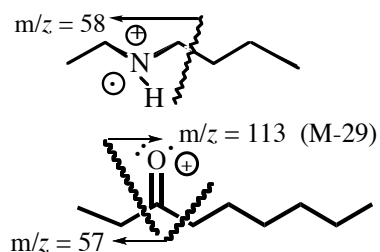
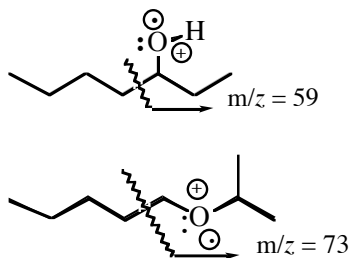
Table 2-7 on p. 103 in Padías gives masses of many common neutral species lost in molecular ion fragmentations.

b. Breakage at Branch Points of Saturated Carbon Chains:

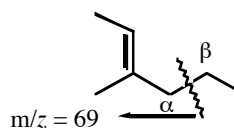
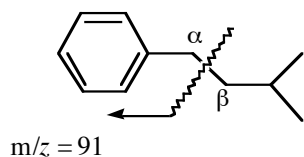
- e.g. The mass spectrum of 2-methylhexane shows a very large peak at $m/z = 43$ due to breakage of the indicated bond.

**c. Breakage of C-C bonds next to the site of attachment of a heteroatom:**

- e.g. The mass spectra of 2-heptanol, N-ethylbutylamine, isopropyl pentyl ether, and 3-nonanone show prominent peaks at 59, 58, 73 and 57 respectively due to the breakages indicated below.

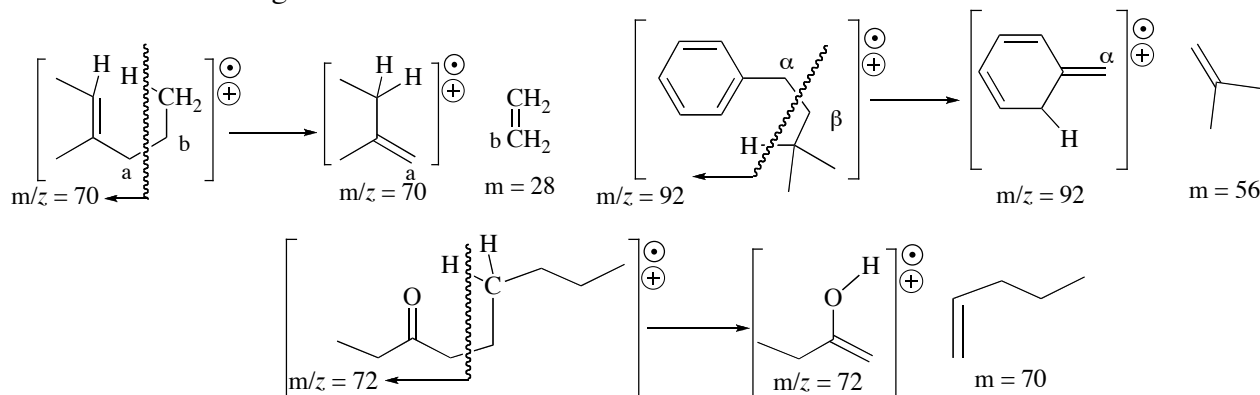
**d. Breakage between atoms that are α & β to $\text{C}=\text{C}$'s or aromatic rings:**

- e.g. The mass spectra of (3-methylbutyl)benzene and (*E*)3-methyl-2-hexene show prominent peaks at 91 and 69 respectively due to the breakages indicated below.



e. Breakage of Unsaturated Compounds with Rearrangement:

e.g. The mass spectra of (*E*)-3-methyl-2-hexene, (3-methylbutyl)benzene and 3-nonanone show a prominent peaks at 70, 92 and 72 respectively due to the breakages indicated below.

**Molecular Formula from the Mass Spectrum.**

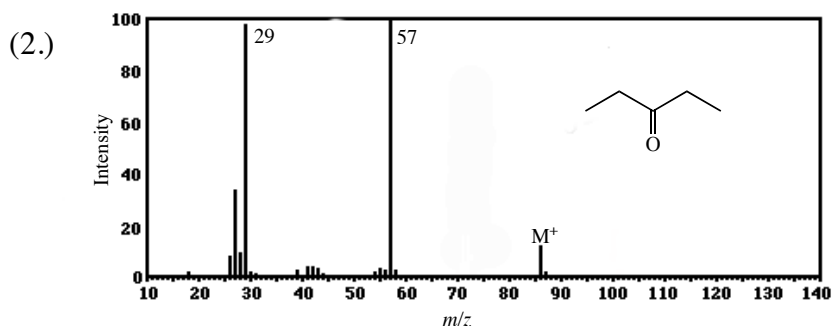
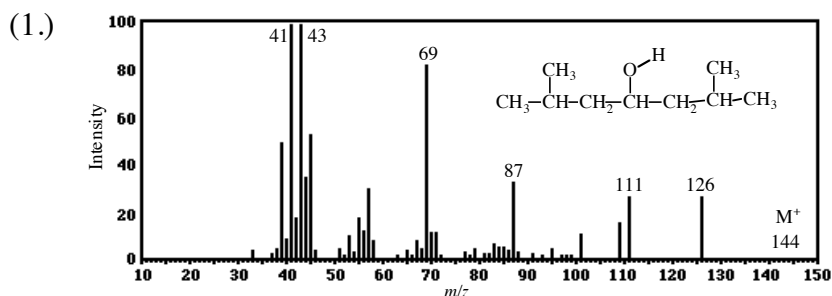
You may have noted that there are often some small peaks at higher mass than expected for the molecular ion. These peaks usually appear at masses of $M + 1$ and $M + 2$. They are caused by the presence of heavy isotopes of carbon, nitrogen and oxygen in some of the molecules. It is possible to use the intensities of the $M + 1$ and $M + 2$ to determine the molecular formula of a compound from the mass spectrum (See CGW pp. 50-51). The in-lab questions for experiment 8 explore this aspect of mass spectrometry.

Additional Out of Class Applications:

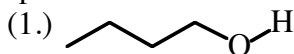
Now use the reference MS information in Padías pp. 106-113 and the activities on pp. D1→D6, to devise responses to the following:

a. Predicting Mass Spectra from Structures:

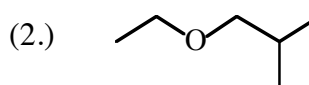
Propose structures for the ions that most likely produced the numbered peaks in each of the following spectra.



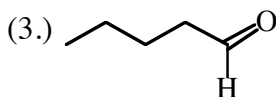
- b. Predict 2 major ion peaks, in addition to the molecular ion, that should appear in the mass spectra of the following compounds.



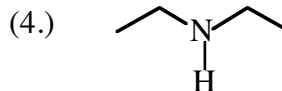
1-butanol



ethyl (1-methylpropyl) ether



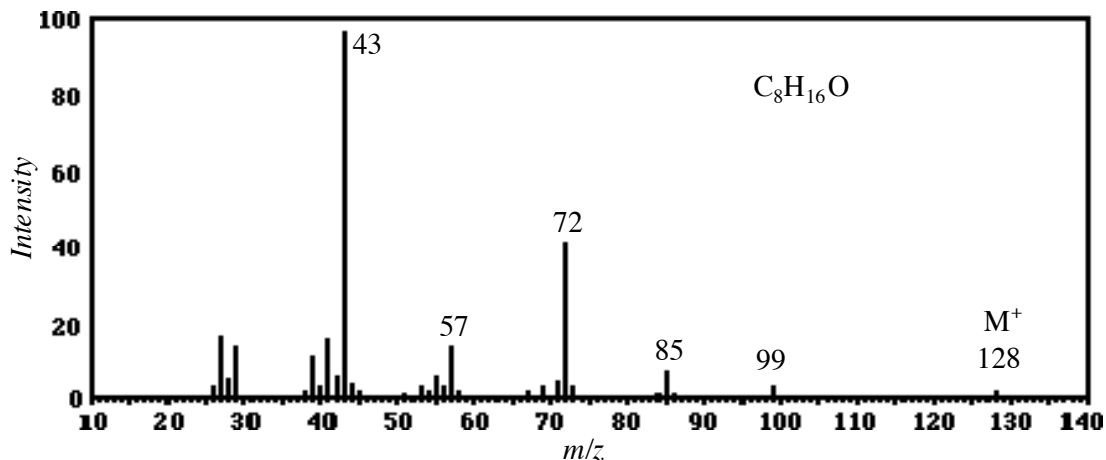
pentanal



diethylamine

- c. Using your experience with mass spectra, identify as many structural groups present in the molecules with the following molecular formulas and mass spectra. Provide your warrant for each structure suggested.

(1.)



(2.) Compound -- Molecular Formula $C_{11}H_{14}O$

Mass Spectrum -- prominent peaks 43, 91, 147 & 162

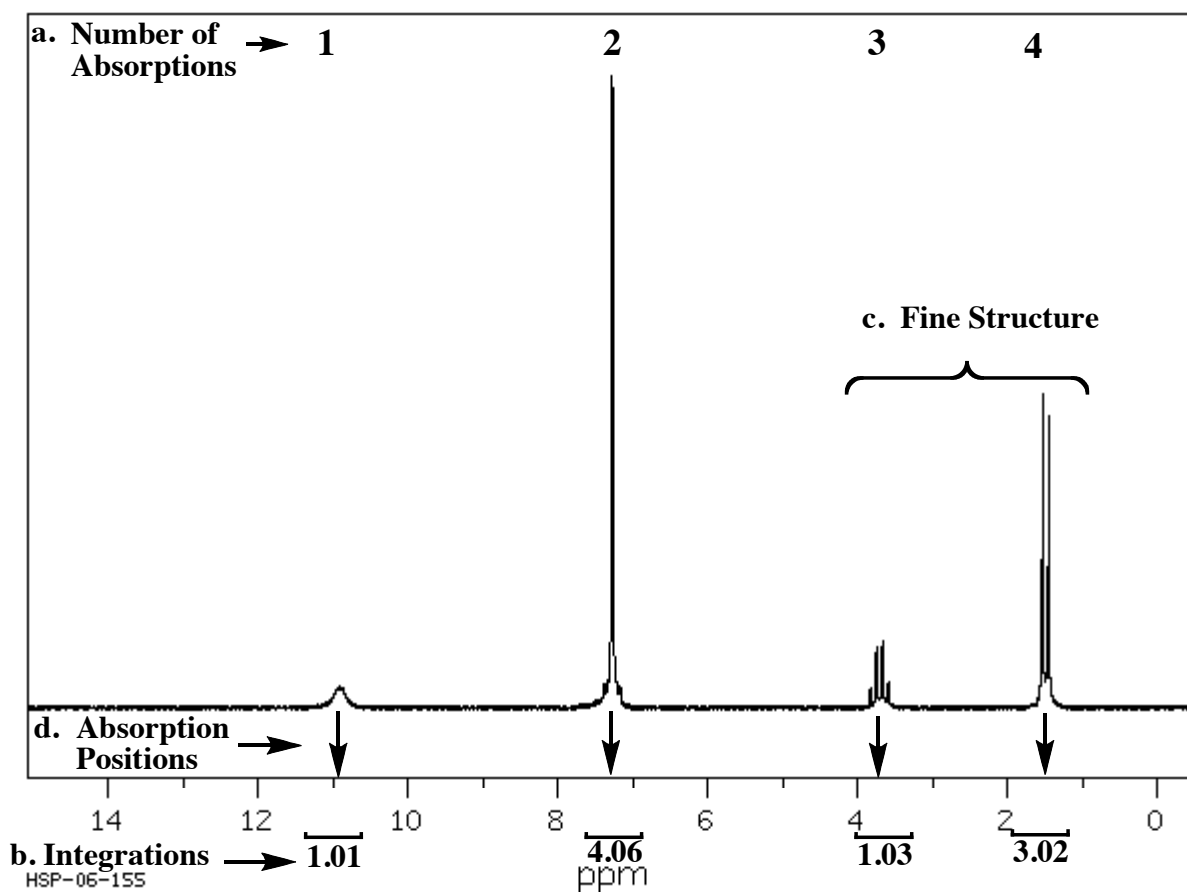
Appendix E. Nuclear Magnetic Resonance Spectroscopy.

Investigation of NMR Spectra:

(See also, Padías pp. 77-81 & CGW pp. 52-62 & 269-301)

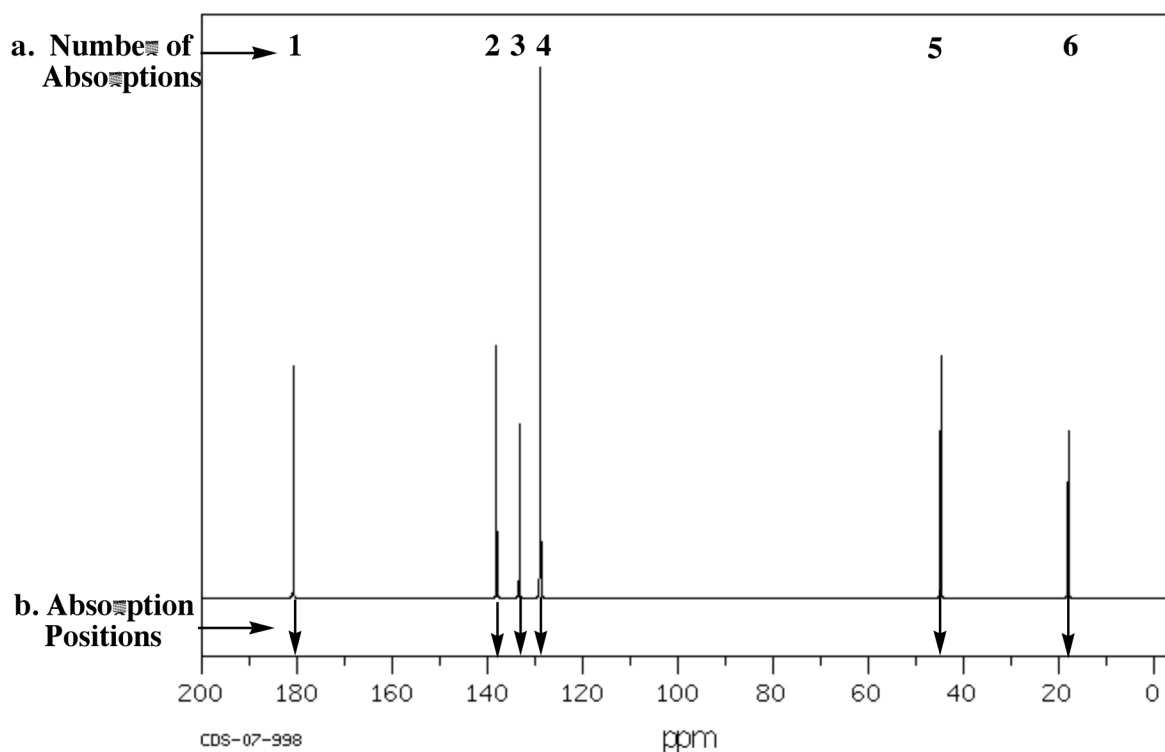
Nuclear Magnetic Resonance Spectra provide information about the environments of various nuclei in molecules. The most useful for organic compounds are protons (^1H) and Carbon (^{13}C). These spectra contain a great deal of information so they can appear to be very complex as illustrated in Figures 1 & 2. However, we can dissect each type of spectrum into its components each of which provides its own specific information. In the next few pages, we will explore the structural features of organic compounds that affect each of these components to see how each spectral component provides information about the molecular structure.

Figure 1: A Proton NMR (^1H -NMR) of an organic compound.



Components of an ^1H -NMR Spectrum:

- Number of Absorptions:** The number of different regions of the spectrum where absorptions (peaks) appear.
- Integrations of the peaks:** Electronically determined areas under each peak in the spectrum.
- Fine Structure of Peaks:** The shape of the peaks. Note that peaks 1 & 2 show a single maximum while peaks 3 & 4 have more than one maximum. Peaks 1 & 2 are termed singlets (one maximum), peak 3 is a quartet (four maxima) and peak 4 is a doublet (two maxima).
- Absorption Positions:** Reported as a number on the abscissa of the NMR graph. The numerical abscissa scale for NMR is called the chemical shift scale and is expressed in units δ . Most proton absorptions appear between 0 & 12 δ . E.g. the chemical shift for peak 3 is 3.85 δ .

Figure 2: A Carbon NMR (^{13}C -NMR) of an organic compound.

Components of a ^{13}C -NMR Spectrum:

- Number of Absorptions:** The number of different regions of the spectrum where absorptions (peaks) appear.
- Absorption Positions:** Reported as a number on abscissa of the NMR graph. As with ^1H -NMR, the numerical abscissa scale for ^{13}C -NMR is called the chemical shift scale. Most carbon absorptions appear between 0 & 200 δ . E.g. the chemical shift for peak 5 is 42.5 δ .

Note: ^{13}C spectra are considerably less complex than ^1H spectra. Because of the differences in the way the spectra are collected, ^{13}C spectra do not provide integration and fine structure information. Also, note that the chemical shift scale for ^{13}C is much wider than that for ^1H spectra.

Number of absorptions and chemical shifts of both ^1H & ^{13}C Spectra

Compound	^{13}C Chemical Shift (δ)	^1H Chemical Shift (δ)
$\text{CH}_3\text{—CH}_3$	7	0.9
$\text{CH}_3\text{—CH}_2\text{—CH}_3$	15, 22	0.9, 1.3
$\text{CH}_3\text{—CH}_2\text{—CH}_2\text{—CH}_3$	14, 25	0.9, 1.3
$\begin{array}{c} \text{CH}_3 \\ \\ \text{CH}_3\text{—C—CH}_3 \\ \\ \text{H} \end{array}$	22, 35	0.9, 1.5
$\begin{array}{c} \text{CH}_3 \qquad \text{CH}_3 \\ \qquad \quad \\ \text{CH}_3\text{—C—CH}_2\text{—C—CH}_3 \\ \qquad \quad \\ \text{H} \qquad \quad \text{H} \end{array}$	23, 25, 48	0.9, 1.3, 1.5
$\begin{array}{c} \text{CH}_3 \\ \\ \text{CH}_3\text{—C—CH}_3 \\ \\ \text{CH}_3 \end{array}$	28, 32	0.9
$\text{CH}_3\text{—Cl}$	29	3.1
$\text{CH}_3\text{—CH}_2\text{—Cl}$	16, 37	1.5, 3.5
$\begin{array}{c} \text{Cl} \\ \\ \text{CH}_3\text{—C—CH}_3 \\ \\ \text{H} \end{array}$	25, 37	1.5, 3.8
$\text{Cl—CH}_2\text{—Cl}$	54	5.3
CHCl_3	82	7.3
$\text{CH}_3\text{—O—CH}_3$	56	3.3
$\text{CH}_3\text{—CH}_2\text{—O—CH}_2\text{—CH}_3$	15, 63	1.0, 3.4
$\begin{array}{c} \text{CH}_3 \qquad \text{CH}_3 \\ \qquad \quad \\ \text{CH}_3\text{—C—O—C—CH}_3 \\ \qquad \quad \\ \text{H} \qquad \quad \text{H} \end{array}$	23, 66	1.0, 3.5
$\text{CH}_3\text{—CH}_2\text{—O—CH}_3$	14, 54, 65	1.0, 3.3, 3.4
$\text{CH}_3\text{—F}$	67	4.3

Table 1: ^1H & ^{13}C Chemical Shifts of NMR Absorptions of Some Organic Compounds.

1. Exploration of number of absorptions:

Note the variations in the numbers of absorptions of the related compounds in Table 1. The data was extracted from Table 1 on p. E3.

$\text{CH}_3\text{---CH}_3$	has one ^{13}C and one ^1H absorption v
$\text{CH}_3\text{---CH}_2\text{---CH}_3$	has two ^{13}C and two ^1H absorption v's
$\text{CH}_3\text{---CH}_2\text{---CH}_2\text{---CH}_3$	has two ^{13}C and two ^1H absorption v's
and	
$\begin{array}{c} \text{CH}_3 \qquad \qquad \text{CH}_3 \\ \qquad \qquad \quad \\ \text{CH}_3\text{---C---CH}_2\text{---C---CH}_3 \\ \qquad \qquad \quad \\ \text{H} \qquad \qquad \quad \text{H} \end{array}$	has three ^{13}C and three ^1H absorption v's
But that:	
$\begin{array}{c} \text{CH}_3 \\ \\ \text{CH}_3\text{---C---CH}_3 \\ \\ \text{CH}_3 \end{array}$	has two ^{13}C and only one ^1H absorption v's
$\text{CH}_3\text{---CH}_2\text{---Cl}$	has two ^{13}C and two ^1H absorption v's
and	
$\text{CH}_3\text{---CH}_2\text{---O---CH}_3$	has three ^{13}C and three ^1H absorption v's

Table 2: Numbers of NMR Absorptions of Some Organic Compounds

2. Invention of a relationship between molecular structure and the number of ^1H & ^{13}C absorptions.

Remembering that NMR absorptions are due to proton (^1H -NMR) and carbon (^{13}C -NMR) nuclei in the molecule, propose a structural correlation for the numbers of absorptions in the NMR spectra illustrated in Table 1 (your claim). Provide your warrant.

3. Exploration of chemical shifts of NMR absorptions:

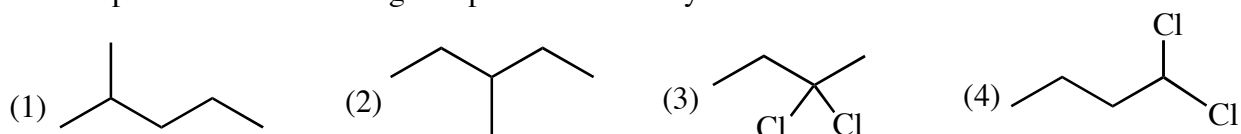
Using the relationship developed in 2, assign the ^{13}C & ^1H chemical shifts in Table 1 to specific carbon atoms or groups of hydrogen atoms in each compound. Illustrate your assignments in Table 1 and provide your warrants.

4. Invention:

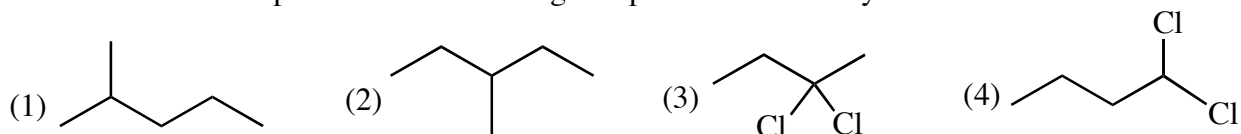
Note that the magnitudes of some of the chemical shifts from the compounds containing oxygen or halogens are different from those for similar atoms in hydrocarbons. Use the patterns of chemical shifts to propose a structural cause of the effect of oxygen and halogens on ^{13}C & ^1H chemical shifts. (your claim) Provide your warrant citing specific data from Table 1 to support it.

5. Applications:

- a. Using your claim in 2, predict the numbers of ^1H & ^{13}C NMR absorptions in the NMR spectra of the following compounds. Provide your warrant for each.



- b. Using your claim in 4, predict approximate chemical shifts of ^1H & ^{13}C NMR absorptions in the NMR spectra of the following compounds. Provide your warrant for each.



Integration (relative areas under the peaks) - ^1H Spectra only

1. Exploration:

Note the variations in the areas of absorptions of the related compounds in Table 3. Using your experience from pp. E 3-E5, assign each relative area in Table 3 to specific groups of hydrogen atoms in each compound. Illustrate assignments in Table 3 and provide your warrant citing data from Tables 1, 2 and/or 3 to support it.

Compound	^1H Chemical Shift (δ)	Rel. Area
$\text{CH}_3\text{---CH}_2\text{---CH}_3$	0.9	3
	1.3	1
$\text{CH}_3\text{---CH}_2\text{---CH}_2\text{---CH}_3$	0.9	3
	1.3	2
$\begin{array}{c} \text{CH}_3 \\ \\ \text{CH}_3\text{---C---CH}_3 \\ \\ \text{H} \end{array}$	0.9	9
	1.5	1
$\begin{array}{c} \text{CH}_3 \qquad \text{CH}_3 \\ \qquad \quad \\ \text{CH}_3\text{---C---CH}_2\text{---C---CH}_3 \\ \qquad \quad \\ \text{H} \qquad \quad \text{H} \end{array}$	0.9	6
	1.3	1
	1.5	1

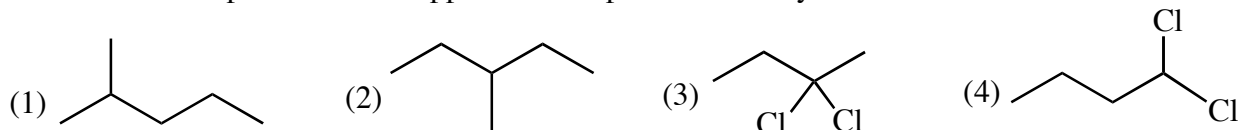
Table 3: Relative Areas of ^1H -NMR Absorptions of Some Organic Compounds

2. Invention

Propose a structural correlation for the magnitudes of the relative areas of absorptions in the NMR spectra in Table 3 (your claim). Provide your warrant.

3. Application:

Using your claim in 2., predict the relative areas of the peaks in the ^1H -NMR spectra of the following compounds. Note that you predicted the number of peaks and their chemical shifts for these compounds in the Application on p. E5. Provide your warrant for each.

***Fine structure of ^1H Peaks:*** (Number of Maxima in Each Absorption)

1. Exploration:

Observe the peaks in the spectra in Figure 3 (E7-E9).

- Use the relationship developed in the activities on pp. E3-E6 to assign the individual peaks in each spectrum to specific groups of protons in the structure.
- Note the presence or absence of multiple maxima in peaks in each spectrum.

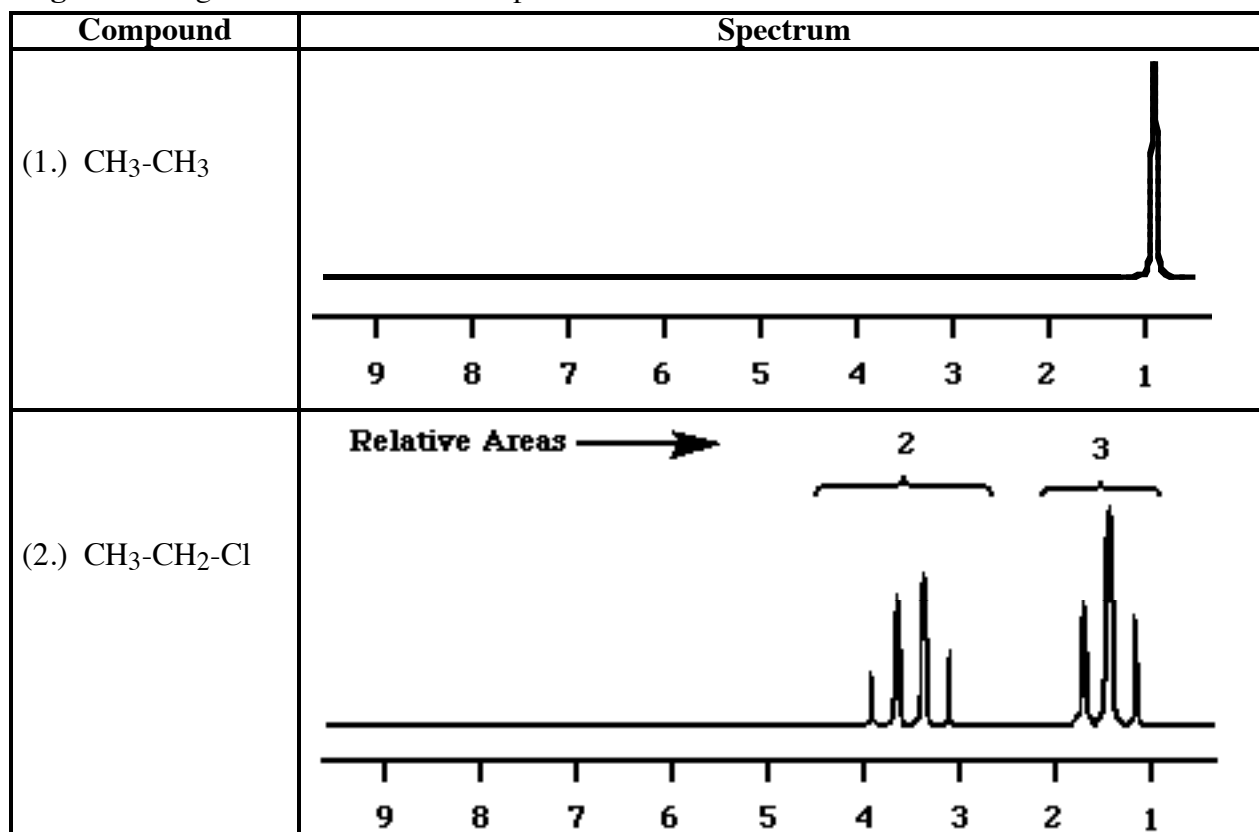
Figure 3: High resolution ^1H -NMR Spectra

Figure 3: High resolution ^1H -NMR Spectra (Continued)

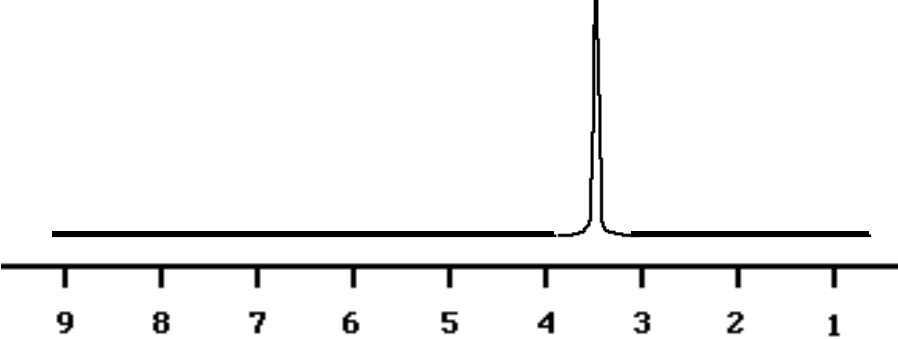
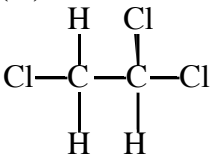
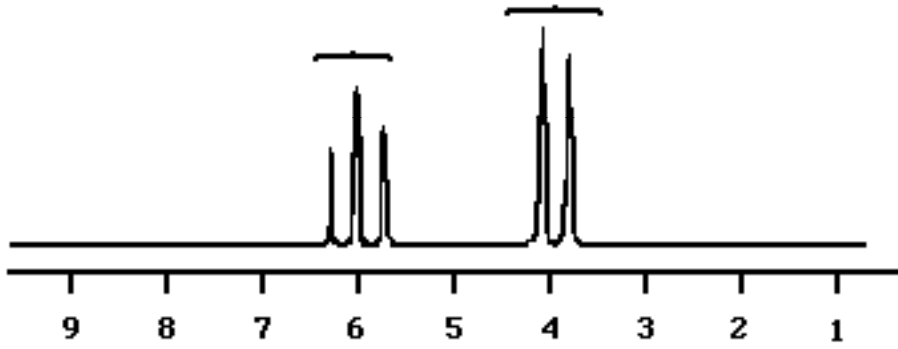
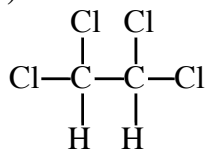
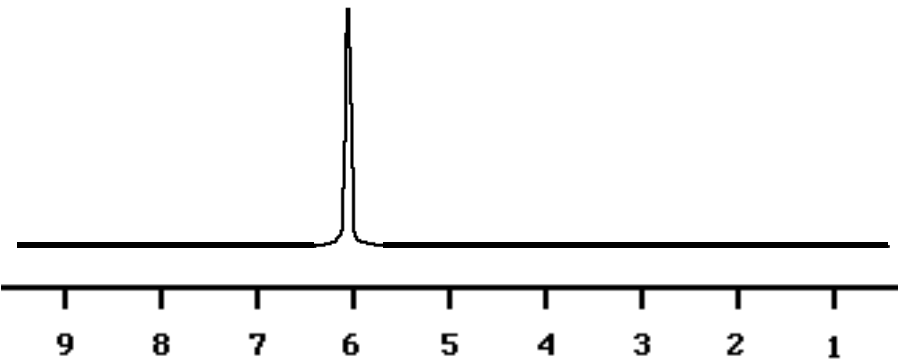
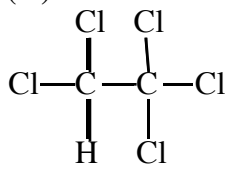
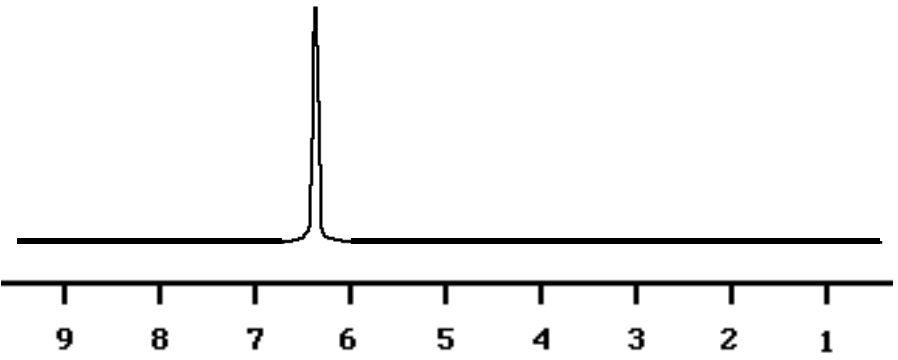
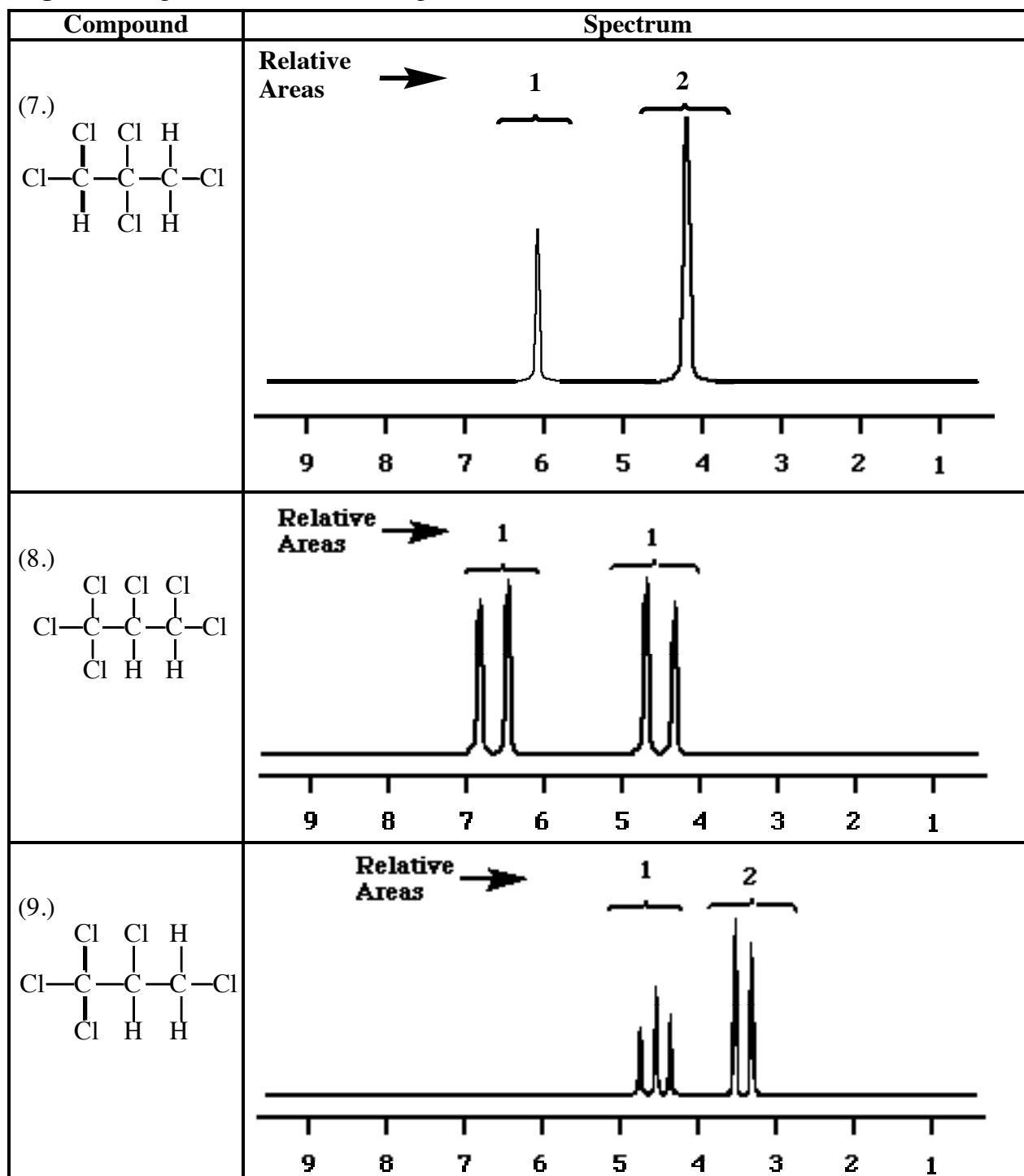
Compound	Spectrum
(3.) $\text{Cl}-\text{CH}_2-\text{CH}_2\text{Cl}$	 The spectrum shows a single sharp peak at approximately 3.5 ppm. The x-axis is labeled from 9 to 1 ppm.
(4.) 	Relative Areas $\rightarrow$ 1 2  The spectrum shows two multiplets. The first multiplet is at approximately 6.1 ppm and the second is at approximately 3.9 ppm. The x-axis is labeled from 9 to 1 ppm.
(5.) 	 The spectrum shows a single sharp peak at approximately 5.9 ppm. The x-axis is labeled from 9 to 1 ppm.
(6.) 	 The spectrum shows a single sharp peak at approximately 5.9 ppm. The x-axis is labeled from 9 to 1 ppm.

Figure 3: High resolution ^1H -NMR Spectra (Continued)

2. Invention;

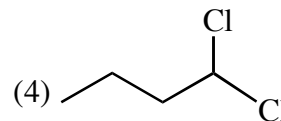
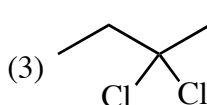
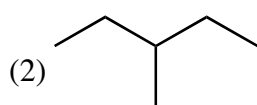
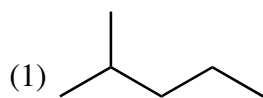
- a. The spectra of compounds (1.), (3.), (5.), & (6.) consist of single unsplit peaks, while those of compounds (2.) & (4.) consist of 2 sets of multiple peaks. What does comparison of these two groups of spectra suggest about a structural requirement for splitting (presence of multiple maxima)? Provide your warrant.

- b. Note also, that while the spectra of compounds (2.), (4.), (8.) & (9.) contain multiple maxima, the spectrum of compound (7.) does not. What additional structural requirement for splitting is suggested by these comparisons? Provide your warrant.

- c. Use your claims from (1.) and (2.) to develop a method for predicting the number of peaks produced in each "split" peak from the structure of the compound. Provide your warrant.

3. Application:

Using your claims in 2., predict the splitting patterns (multiplicity) of the peaks in the ^1H -NMR spectra of the following compounds. Note that you predicted the number of absorptions and relative areas of peaks for these compounds in the applications on pp. E5-E7. Provide your warrant for each multiplicity (splitting) assignment.



Characteristics of Spin-Spin Coupling (Splitting).

Note: That in Figure 3 (E7-E9), Cpds. (2.), (4.), (8.) & (9.), both sets of "coupled" protons are split by the same amount (# of Hz = J_{AX} the coupling constant).

Further Consideration Absorption Positions (Chemical Shifts) - ^1H & ^{13}C Spectra**1. Alkene Chemical Shifts:****a. Invention:**

Considering your claim on the structural effects on chemical shifts of nuclei in the saturated compounds in the activities on pp. E4-E5, provide an explanation of following chemical shift data from alkenes on the basis of changes in electron distribution in the σ -bonds of the alkenes in Table 4 (E10-E11) and on the following page.

Table 4: Alkene Chemical Shifts:

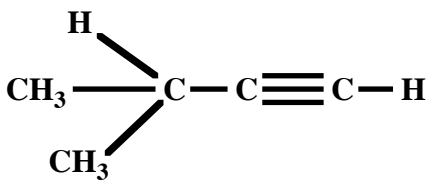
Compounds	^{13}C ν (δ)	^1H ν (δ)
$\text{CH}_2=\text{CH}_2$	123	4.2
$\text{CH}_3-\text{CH}=\text{CH}_2$	17, 116, 133	1.5, 4.6, 5.1
$\text{CH}_3-\text{CH}_2-\text{CH}=\text{CH}_2$	14, 26, 115, 137	1.0, 2.0, 4.6, 5.1
$\begin{array}{c} \text{CH}_3 \\ \diagdown \\ \text{C}=\text{CH}_2 \\ \diagup \\ \text{CH}_3 \end{array}$	25, 108, 142	1.6, 4.6
$\begin{array}{c} \text{CH}_3 \quad \text{CH}_3 \\ \diagdown \quad \diagup \\ \text{C}=\text{C} \\ \diagup \quad \diagdown \\ \text{CH}_3 \quad \text{H} \end{array}$	19, 25, 118, 135	1.5, 1.6, 5.1
$\begin{array}{c} \text{CH}_3 \quad \text{CH}_3 \\ \diagdown \quad \diagup \\ \text{C}=\text{C} \\ \diagup \quad \diagdown \\ \text{CH}_3 \quad \text{CH}_3 \end{array}$	19, 127	1.6
$\begin{array}{c} \text{H} \\ \diagup \\ \text{CH}_3 \quad \text{C} \quad \text{C}=\text{CH}_2 \\ \diagdown \quad \diagup \\ \text{CH}_3 \quad \text{H} \end{array}$	22, 32, 111, 142	1.0, 2.6, 4.6, 5.1
$\begin{array}{c} \text{CH}_3 \quad \text{CH}_3 \\ \diagdown \quad \diagup \\ \text{C}=\text{C} \\ \diagup \quad \diagdown \\ \text{H} \quad \text{H} \end{array}$	11, 125	1.5, 5.1

b. Alkynes:

Exploration:

Compare the ^{13}C & ^1H chemical shifts of the alkynes in Table 5 with those of corresponding alkenes in Table 4. How do the effects of carbon-carbon triple bonds on ^{13}C & ^1H chemical shifts of other atoms in the structures compare with those in alkenes?

Table 5: Alkyne Chemical Shifts:

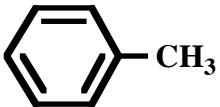
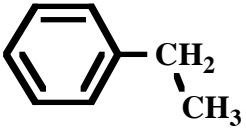
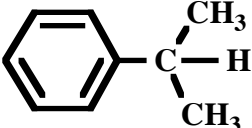
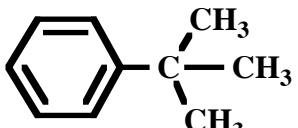
Compounds	^{13}C ν (δ)	^1H ν (δ)
$\text{H}-\text{C}\equiv\text{C}-\text{H}$	72	2.4
$\text{CH}_3-\text{C}\equiv\text{C}-\text{H}$	4, 68, 80	1.7, 2.1
$\text{CH}_3-\text{C}\equiv\text{C}-\text{CH}_3$	1, 76	1.7
$\text{CH}_3-\text{CH}_2-\text{C}\equiv\text{C}-\text{H}$	12, 13, 67, 86	1.1, 1.8, 2.1
	21, 22, 68, 89	1.1, 1.9, 2.1

c. Aromatic Compounds

Exploration:

How do the data in Table 6 either support or conflict with the patterns discovered from Tables 4 & 5 (E11 & E12)? Provide your warrant.

Table 6: Aromatic Compound Chemical Shifts:

Compounds	^{13}C ν (δ)	^1H ν (δ)
	129	7.2
	21, 126, 128, 129, 138	2.4, 7.2
	16, 29, 126, 127, 128, 140	1.3, 2.7, 7.2
	24, 33, 126, 127, 128, 149	1.3, 2.9, 7.2
	31, 35, 125, 126, 128, 147	1.3, 7.2

d. Carbonyl Compounds**Table 7: Carbonyl Compound Chemical Shifts:**

Compounds	^{13}C ν (δ)	^1H ν (δ)
	27, 200	2.2, 9.8
	6, 37, 200	1.1, 2.4, 9.7
	27, 206	2.1
	7, 24, 36, 207	1.1, 2.1, 2.5
	16, 22, 45, 210	1.1, 2.1, 2.6
	52, 161	3.8, 8.1
	17, 50, 171	2.0, 3.7
	9, 26, 50, 172	1.1, 2.3, 3.6
	17, 35, 50, 174	1.1, 2.5, 3.6
	14, 17, 59, 171	1.2, 2.0, 4.0
	18, 21, 68, 171	1.2, 2.0, 5.0
	20, 176	2.1, 11.4
	128, 130, 131, 134, 172	7.8, 12.8

Tables 8 & 9 and **Figures 4 & 5** on the next three pages, summarize the outcomes of our discussions. They can be used along with tables in Padías for NMR analysis of organic molecules. Focus particularly on the “Comments” column to see how effects interact.

Chemical Shift Tables:

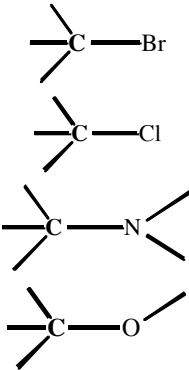
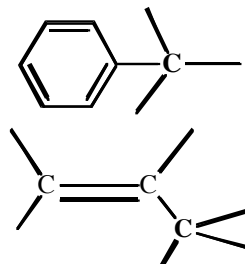
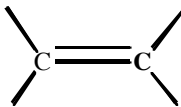
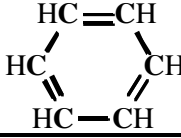
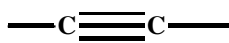
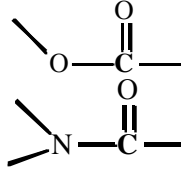
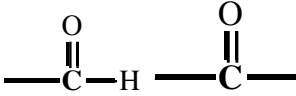
Type of Carbon	Structure	^{13}C δ	Comments
saturated carbon atoms	1° R-CH_3 $2^\circ \text{ R-CH}_2\text{-R}$ $3^\circ \text{ R}_3\text{CH}$	$0 \rightarrow 40$ $10 \rightarrow 50$ $15 \rightarrow 50$	Know that these chemical shifts are $0 \rightarrow 60$ and increase with carbon substitution.
carbons attached to Heteroatoms		$10 \rightarrow 25$ $15 \rightarrow 30$ $30 \rightarrow 45$ $45 \rightarrow 75$	Shifted $\sim 10\text{--}40$ δ down field from the corresponding sat'd hydrocarbon and the extent of the shift increases with the electronegativity of the heteroatom.
benzylic allylic		$\sim 20 \rightarrow 80$ $\sim 20 \rightarrow 80$	Know that these are similar and are shifted ~ 20 δ downfield from sat'd compound
vinyllic		$80 \rightarrow 145$	Shifted ~ 80 δ down field from sat'd carbon
aromatic		$110 \rightarrow 170$	Shifted $\sim 100 \rightarrow 110$ δ down field from sat'd carbon
acetylenic		$65 \rightarrow 95$	Shifted $\sim 50 \rightarrow 60$ δ down field from sat'd carbon
carbonyl carbon atoms of esters and amides		$\sim 155 \rightarrow 185$ $\sim 155 \rightarrow 185$	Know that these are all similar and are generally >150 δ and <190 δ
carbonyl carbon atoms of aldehydes and ketones		$\sim 190 \rightarrow 220$	Know that these are similar and are generally >190 δ

Table 8: Characteristic ^{13}C Chemical Shifts

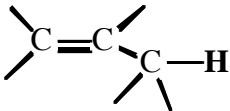
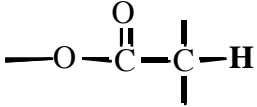
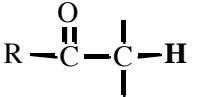
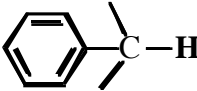
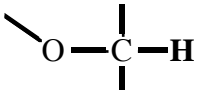
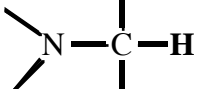
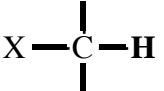
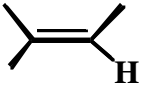
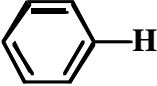
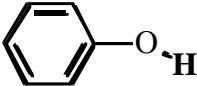
Type of Proton	Structure	^1H δ	Comments
saturated carbon atoms	1° R-CH_3 $2^\circ \text{ R-CH}_2\text{-R}$ $3^\circ \text{ R}_3\text{CH}$	0.9 1.3 1.5	Know the values of these three "Benchmark" frequencies
allylic		~ 1.7	Shifted 0.5 δ downfield from saturated carbon
α to carbonyl groups	 	$2.0 \rightarrow 2.2$ $2.0 \rightarrow 2.7$	Know that these are shifted ~ 1.0 δ downfield from saturated carbon
benzylic		$2.2 \rightarrow 3.0$	Shifted 0.7 $\rightarrow$ 1.5 δ downfield from saturated carbon.
α to oxygen or nitrogen or halogen	  	$3.3 \rightarrow 4.0$ $2.2 \rightarrow 2.5$ $2.0 \rightarrow 4.0$	Know that these are shifted 2.0 $\rightarrow$ 3.0 δ downfield from saturated carbon position.
acetylenic	$\text{—C}\equiv\text{C—H}$	$2.0 \rightarrow 3.0$	Shifted 0.5 $\rightarrow$ 1.5 δ downfield from saturated carbon.
vinylic		$4.6 \rightarrow 5.9$	Shifted $\sim 3 \rightarrow 4$ δ downfield from saturated carbon
aromatic		$6.0 \rightarrow 8.5$	Know
aldehydes	$\text{R—C}(=\text{O})\text{—H}$	$9.0 \rightarrow 10.0$	Know
phenols		$8.5 \rightarrow 9.5$	When H-bonding is restricted, the δ value may be lower.
carboxylic Acids	$\text{R—C}(=\text{O})\text{—OH}$	$10.0 \rightarrow 12.0$	Know that these are $> 10.0\delta$
other protons attached to other heteroatoms	—O—H —N—H	$2.0 \rightarrow 5.0$	Variable or broad due to differences in solvent or hydrogen bonding; These are usually assigned after the C-H protons.

Table 9: Characteristic ^1H Chemical Shifts

Comparison of Carbon and Proton Chemical Shifts

Figure 4

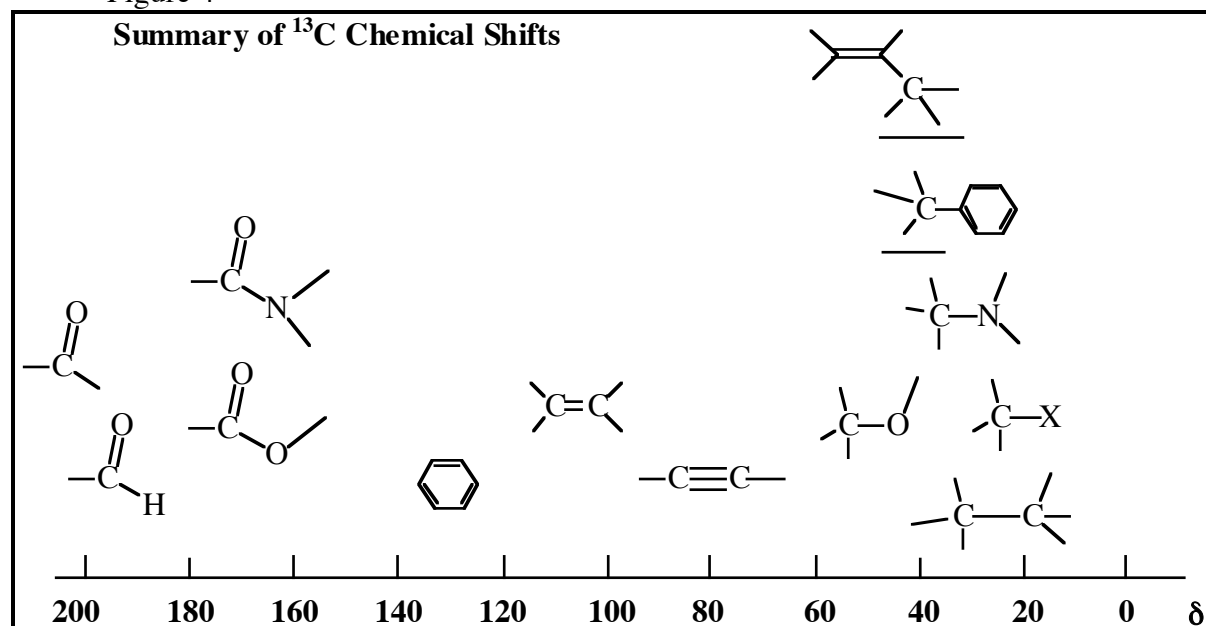
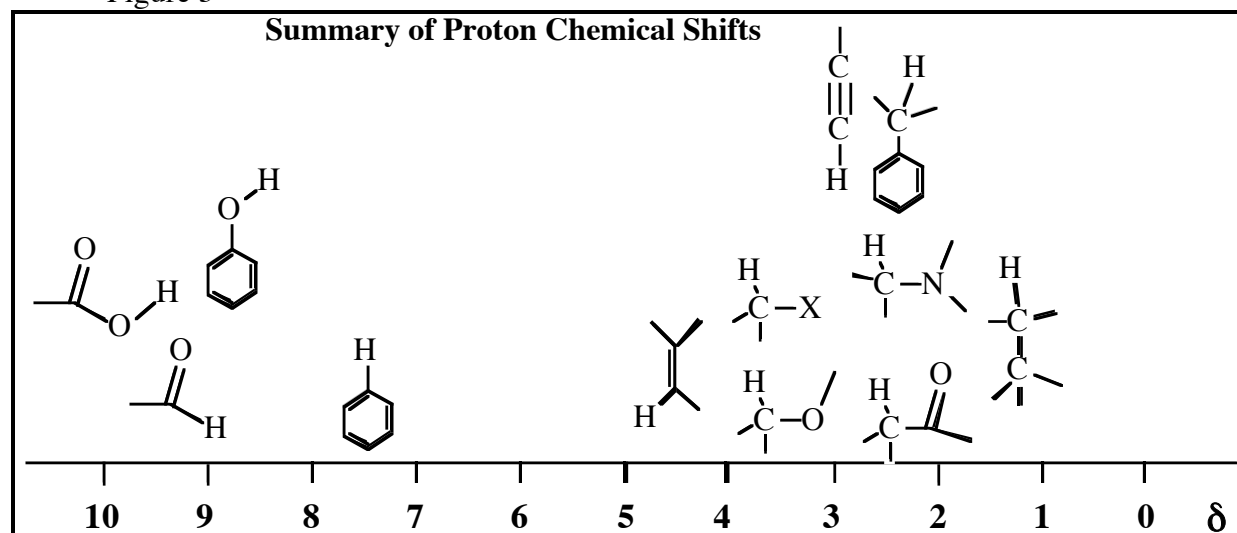


Figure 5



(See Also: CGW pp. 285-295)

- (1.) Splitting is caused by the effects of the magnetic field produced by one magnetic nucleus (in this case a proton) on the magnetic environments of other nuclei in the molecule.
- (2.) Protons are only affected by other **magnetically non-equivalent protons** (protons with different δ values). Thus, compounds such as Figure 3: (1.), (3.), (5.), & (6.) in which all of the protons are indistinguishable (equivalent) yield one peak that is not split, while spectra from compounds such as Figure 3: (2.) & (4.) with non-equivalent groups of protons often exhibit splitting.
- (3.) Although long-range splitting is possible in some situations, **the major splitting (referred to as first order splitting) occurs only among protons that are attached to adjacent carbon atoms.** Thus, Figure 3: compound (7.) yields two singlets (unsplit peaks) since its non-equivalent protons are separated by a CCl_2 group. Thus there are no protons on adjacent carbon atoms. However, the spectra of very similar compounds Figure: (2.), (4.), (8.) & (9.) show splitting because their non-equivalent protons are on adjacent carbon atoms.

NOTE: Because protons on oxygen and nitrogen atoms tend to be acidic and are thus transferred from one molecule to another while the NMR spectrum is being collected, usually they do not split adjacent protons nor are they split by adjacent protons.

In general:

Protons split by n equivalent adjacent nuclei yield $(2nI + 1)$ peaks, where I is the nuclear spin quantum number of the adjacent nucleus. For protons $I = \frac{1}{2}$. So:

For Splitting by Protons Peak Multiplicity = $n + 1$

Where $n = \#$ of equivalent protons on adjacent carbon atoms.

c. Relative Intensities of peaks within a multiplet are given by the coefficients of the binomial expansion $(X + Y)^n$, where n = the number of adjacent equivalent protons. These coefficients can be determined using Pascal's Triangle.

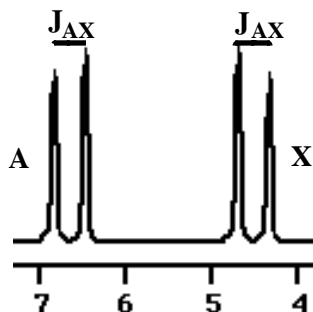
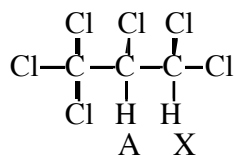
Pascal's Triangle

n							1						
1							1		1				
2						1		2		1			
3					1		3		3		1		
4				1		4		6		4		1	
5			1		5		10		10		5		1

d. Examples:

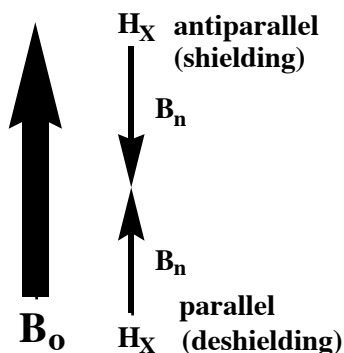
(1.) Consider Figure 3: compound (8.) on E8

- Why are there 4 peaks for only 2 protons?



- Explanation:

Proton A experiences an effective magnetic field, B_{eff} , which is made up of B_0 (the external field), B_{el} (the field produced by proton A's local environment) and B_n (the field produced by proton X).



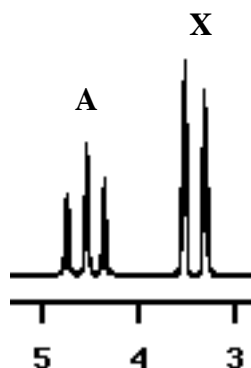
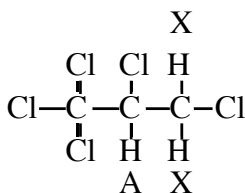
However, B_n has 2 probable orientations with respect to B_0 .

$\therefore \sim \frac{1}{2}$ of the H_A 's experience the B_n of H_X parallel to B_0 (deshielding) and $\sim \frac{1}{2}$ of H_A 's experience the B_n of H_X anti-parallel to B_0 (shielding). So half of the H_A 's absorb at a slightly lower frequency and the other half at a slightly higher frequency.

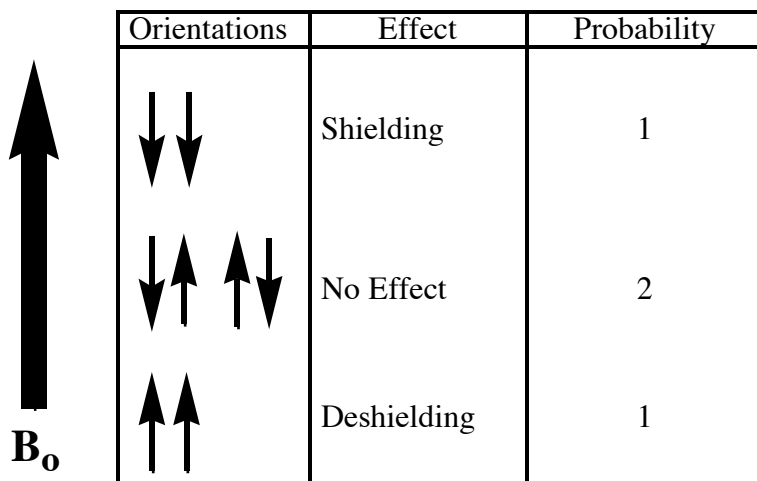
Proton spin orientation diagram

(2.) Consider Figure 3: Compound (9.) on E8

- Why are there 3 peaks for proton A?



- In this case there are 2 X protons and they can produce 3 different types of orientations with respect to B_0 .



Orientations	Effect	Probability
	Shielding	1
	No Effect	2
	Deshielding	1

Proton spin orientation diagram

$\therefore$ H_A exhibits a triplet (3 peaks)
with a total area = 1 proton
with relative areas of 1:2:1

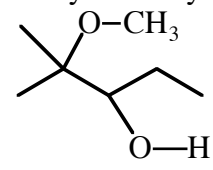
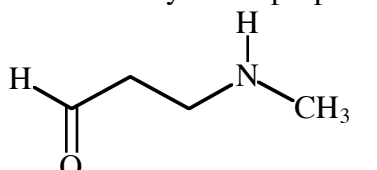
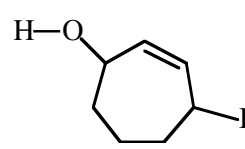
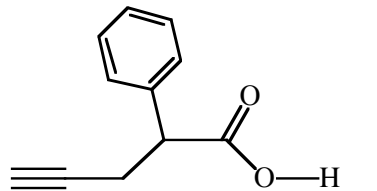
H_X exhibits a doublet (2 peaks)
with a total area = 2 proton
with relative areas of 1:1

Out of Class Applications on NMR:

Now using the your experience with the NMR activities, the reference NMR information In Tables 8 & 9, Figures 4 & 5 of the lab manual, CGW pp. 285-295 devise responses to the following:

For each of the following compounds:

- Predict the number of different chemical shift signals that would be expected in its 1H and ^{13}C NMR spectra,
- Predict the approximate chemical shift value expected for each signal.
- Indicate which nucleus is responsible for each signal.

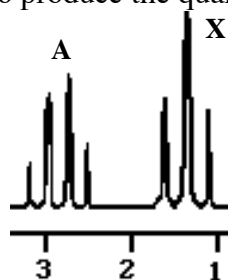
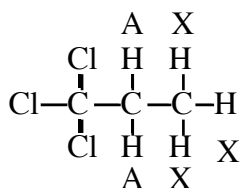
(1.) 2-methoxy-2-methyl-pentanol 	(2.) 3-methylaminopropanal 
(3.) 4-iodocyclohept-2-ene-1-ol 	(4.) 2-phenyl-5-hexynoic acid 

- d. Propose a reasonable structure of a compound with the molecular formula $C_6H_{10}O$ and the following NMR chemical shift signals:

1H		^{13}C	
δ	Rel. Area	δ	
0.98	3	14	
1.54	2	21	
2.19	2	22	
2.90	1	51	
4.25	2	79	
		85	

Provide your warrant.

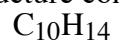
- e. Propose a structure for a molecule with a molecular formula of C_7H_{16} and a ^{13}C NMR spectrum with only three peaks. Then predict the δ values of the three peaks expected from your structure. Provide your warrant. What is the name of the compound you proposed?
- f. 1,1,1-Trichloropropane yields the NMR spectrum indicated below. The triplet produced for protons **X** can be explained using a proton spin orientation diagram similar to that used in the summary of discussion on spin-spin splitting for the triplet produced by protons A in Figure 3: compound (9.). (p. E19) The $(2nI + 1)$ approach (p. E19) correctly predicts that protons A should yield a quartet. But why are there 4 peaks for protons A? Use a proton spin orientation diagram (See pp. E 18-E19) to explain how the X protons can generate the four different magnetic environments required to produce the quartet for the adjacent A protons.



- g. Predict the number of different chemical shift values and the multiplicity of splitting for each peak for each of the following compounds.



h. Give a structure consistent with the following set of data:



^1H		
δ	Rel. Area	Multiplicity
0.90	6	Doublet
1.86	1	Multiplet
2.47	2	Doublet
7.18	5	Singlet

^{13}C	
δ	
22	
30	
45	
126	
128	
129	
142	

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