Long Island University, Brooklyn

Department of Chemistry and Biochemistry

Laboratory Manual,

Fourth Edition

Organic Chemistry

Chemistry 121 and 122

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Chemistry 121

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LABORATORY REGULATIONS

There is NO SMOKING allowed in chemistry laboratories as demanded by fire regulations. NO FOOD is allowed to be brought in or to be eaten in chemistry laboratories.

Goggles or eyeglasses with shatterproof lenses are to be worn at all times as required by law in the State of New York. Anyone without such eye protection WILL NOT be allowed to work in the laboratory!

You must also be appropriately dressed for the laboratory. This means that you r legs , arms, hands, and feet should be covered . NO shorts and no open-toed sandals are permitted. A laboratory coat should be worn and also protective gloves should be used. These are provided by the stockroom on an individual basis. A pair of dishwashing gloves, available at any grocery market, are also suitable. They are inexpensive, provide good protection against most chemicals and will last an entire semester.

Laboratory manuals and textbooks should be read carefully before proceeding with the experiment. Lack of preparation and hastiness can lead to serious accidents in the laboratory. Know the location of all fire extinguishers, fire blankets, eye-washes and emergency showers.

All equipment should be carefully secured and checked before proceeding with the experiment. This will prevent accidents and the need to replace expensive organic glassware. If you do not understand any part of the experiment, please consult with your instructor or your teaching assistant.

Clean your glassware at the end of each lab. Residues can harden and make cleaning very difficult at a later time. In that event, acetone or KOH/methanol will be provided for cleaning your glassware.

Reagents and other chemicals are to be returned to the preparation area so that others can use them as well.

Please do not throw solid waste materials such as boiling stones, matches, broken glass, paper towels, etc. into the sink or drainage areas.

Please do not throw broken glass into the general waste containers but use the special broken glass container for this purpose and please do not throw non-glass items in the broken glass containers.

All organic waste must be deposited in the specially marked bottles in the front hood. DO NOT pour water insoluble material into the sinks and DO NOT pour acids or bases into the organic waste containers.

Laboratory Notebook and Grading

With oversight from the professor, the laboratory Teaching Assistant assigns the laboratory total of 200 points. Each of the ten experiments is worth 20 points: 10 points for the lab write-up in the notebook and 10 points for the Organic Unknown Report Sheet or Yield Report Sheet. A sewn-through, composition-type book must be used for the laboratory notebook and pages should not be removed from it. Please do not use a spiral notebook. This is the working record of what you are going to do in the lab and what you have actually done and observed. The notebook MUST BE PREPARED IN ADVANCE for each experiment so that when you arrive in lab you can perform the experiment directly from your lab notebook and so that YOU CAN RECORD YOUR OBSERVATIONS AND DATA DIRECTLY IN YOUR NOTEBOOK.

POINTS WILL BE DEDUCTED IF YOUR LABORATORY PROCEDURE SECTION IS NOT PREPARED AHEAD OF TIME.

A good notebook should consist of the following:

I Table of Contents - First two pages.

II Numbered pages

III Experimental Write-up

a. Heading: Experiment Title and date.

b. Purpose of the experiment. This is simply a brief, one sentence description of the experiment. For example, the purpose of the first experiment (Melting Points): "To learn the technique of taking melting points and to determine the identity of an unknown using the mixed melting point technique."

c. Preparation Experiments (We don't always prepare a compound, so the exact format may not apply in each case. Use your judgment. Ask your instructor.)

1. Show the overall chemical reaction.
2. Table of physical constants for the materials used and the products. You should record: grams used, molecular weight, number of moles, density, b. p.; use the format given in the procedure for the preparation of *t*-butyl chloride.
3. Note any unusual warnings or cautions. This may be amended during the recitation hour.
4. Diagram of the experimental set-up. This is not always appropriate, depending on the experiment. Usually, the exact nature of the experimental set-up will be discussed immediately prior to the lab in the recitation hour and this section can be completed at that time.
5. Concise, step-by-step procedure to be followed in the lab. This is your working guide for the experiment and it must be detailed enough so that you can perform the experiment directly from this outline. It is useful to number the steps and to leave plenty of space for making additional notes during the recitation hour.
6. Experimental observations. **These should be added as the experiment is being performed.** These observations often deal with color changes, temperatures at which your compound boiled or distilled, etc. Describe the color and appearance of your product (i.e. "yellow, crystalline solid"; "white powder"; "cloudy, pale yellow liquid", etc.). Record the melting point range for solids and boiling point range for distilled products. Record any changes you made to the procedure for whatever reason. Be honest. Record what actually happened, not what was supposed to happen. You are still learning in the laboratory and many things do go wrong, even for the experienced chemist. You are not being judged solely your results but on your efforts and the accuracy of your observations.

6. Conclusions. Briefly discuss your results. If the yield was low try to explain why. ALL CALCULATIONS INCLUDING % YIELD CALCULATIONS MUST BE INCLUDED IN THE NOTEBOOK as part of your conclusions.

Sample Percent Yield Calculation

Percent yield is defined as:

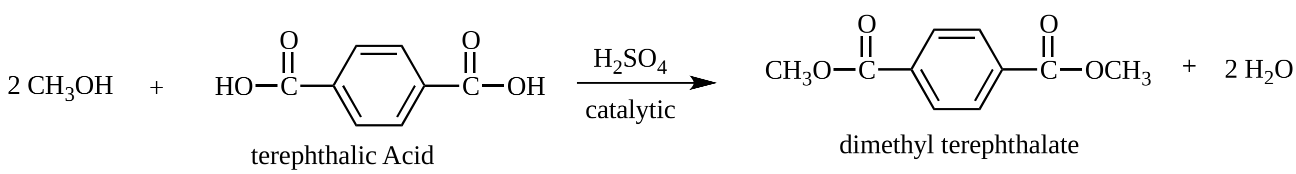
Percent yield = (actual yield/theoretical yield) x 100.

The actual yield is the number in grams of the product that you actually isolate at the end of the experiment.

The theoretical yield is the number of moles of the limiting reagent times the molecular weight of the product. The limiting reagent is defined as the reagent that is present in the smallest number of equivalents used in the reaction. It is almost always the organic starting material and the number of equivalents is simply the number of moles used divided by the stoichiometric coefficient.

Sample Calculation. For the reaction shown in Figure 2.4, two equivalents of methanol

Figure 3.4



react with one equivalent of terephthalic acid to give one equivalent of dimethyl terephthalate. So, for every mole of terephthalic acid consumed, two moles of methanol are also used up. The catalyst, H_2SO_4, is not consumed in the reaction and therefore it does not have to be considered when determining the limiting reagent. To find the number of equivalents, simply divide the number of moles by the stoichiometric coefficient.

For example, if we do a reaction in which we start with 1.0 g of terephthalic acid and use 5 mL of methanol and 3 drops of sulfuric acid and isolate 0.65 g. of product, the table of reagents that summarizes the information is given below.

Physical Constants

Compound	Mol. Wt (g/mol)	Amt used	Density (g/mL)	Wt used(g)	Moles used	Equiv. used
Terephthalic acid	166.14	1.0 g	solid	1.0	0.00602	0.00602
Methanol	32.04	5 mL	0.7914	3.96	0.124	0.0620
Sulfuric acid	98.08	3 drops	1.84	0.2	Not used up	Not used up

The smallest number of reaction equivalents is 0.00602 for terephthalic acid. It is therefore the limiting reagent.

Therefore: theoretical yield = moles limiting reagent x molecular weight of product
= 0.00602 mol x 194.19 g/mol = 1.17 g

Therefore, the Percent yield = (0.65 g/1.17 g) x 100 = 56%.

Another way to calculate the percent yield is to divide moles of product by moles of limiting reagent used.

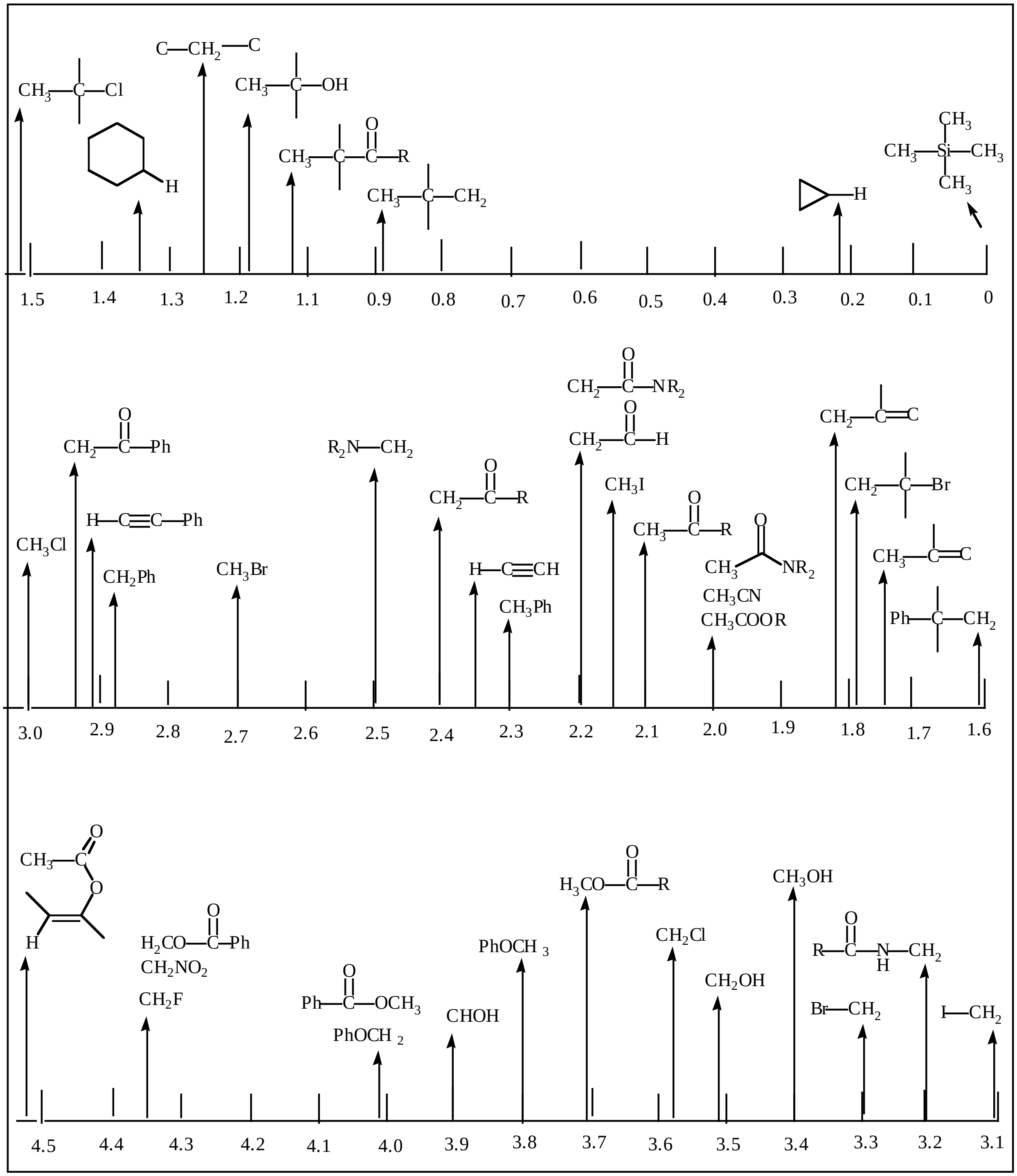
Moles Product = (0.65 g)/ (194.19 g/mol) = 0.00347 mol
Therefore, % yield = (0.00347 mol/0.00602 mol) x 100 = 56%

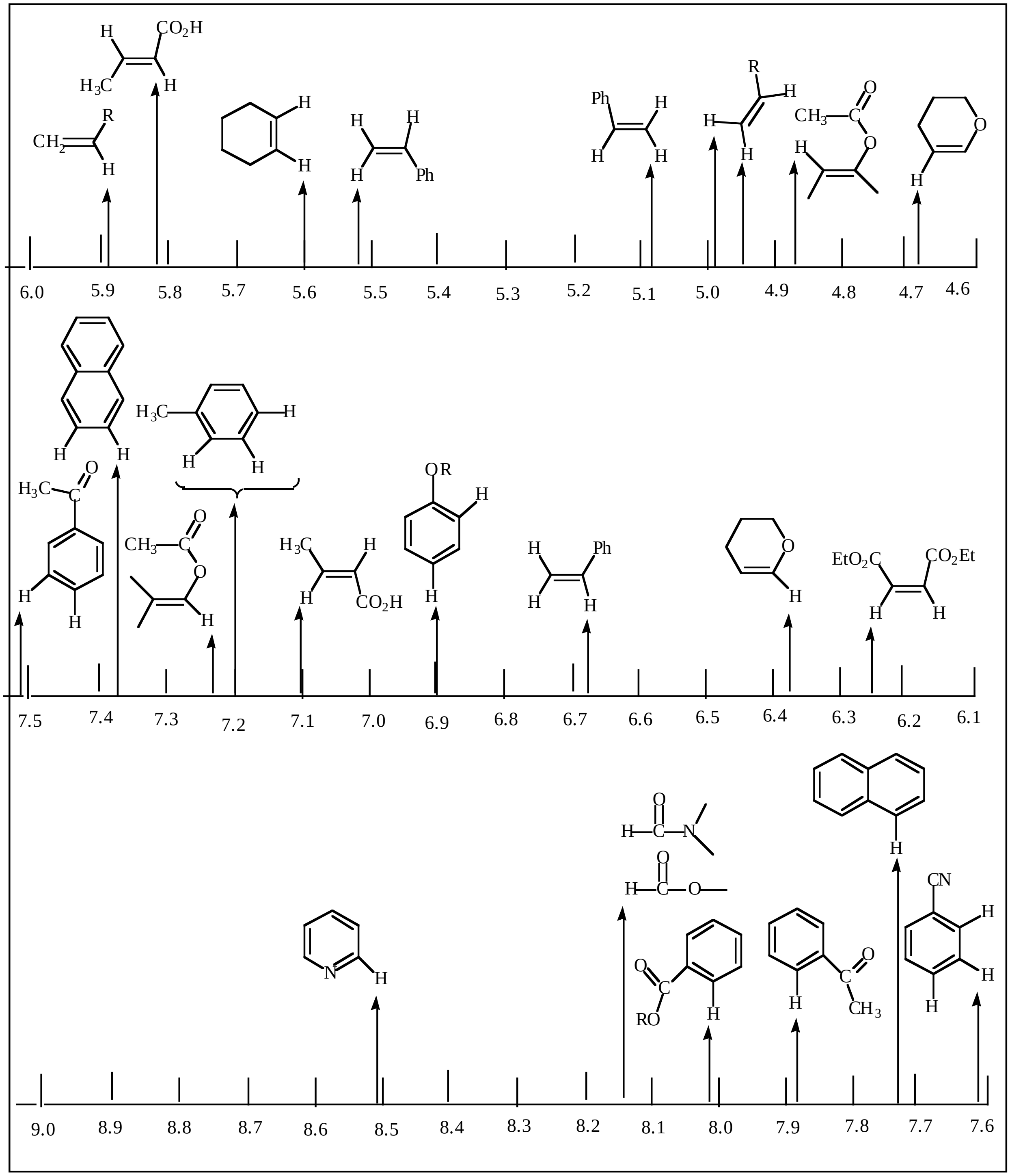
Characteristic Infrared Absorption Frequencies

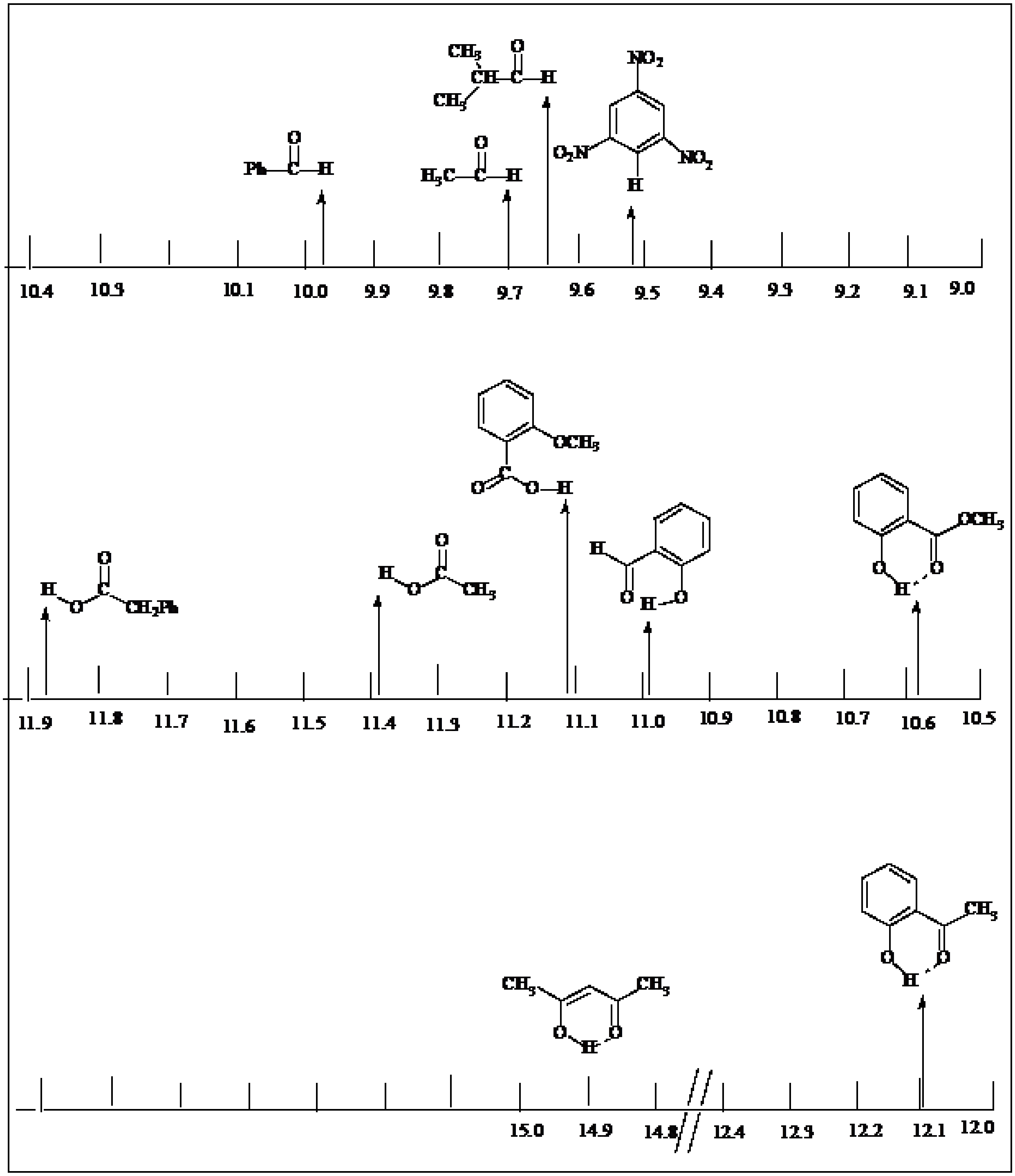
Type of Vibration		Frequency (cm^{-1})	Intensity
C-H			
Alkanes	(stretch)	2980 - 2850	s
$-CH_3$	(bend)	1450 and 1375	m
$-CH_2-$	(bend)	1465	m
Alkenes	(stretch)	3100-3000	m
	(out-of-plane bend)	1000-650	s
Aromatics	(stretch)	3150-3050	s
	(out-of-plane bend)	900-690	s
Alkyne	(stretch)	ca. 3300	s
Aldehyde		2900-2800	w
		2800-2700	m
C-C			
Alkane		Not useful	
C=C			
Alkene		1680-1600	m-w
Aromatic		1600 and 1475	m-w
C,C triple bond			
Alkyne		2250-2100	m-w
C=O			
Aldehyde		1740-1720	s
Ketone		1725-1705	s
Carboxylic acid		1725-1700	s
Ester		1750-1730	s
Amide		1680-1630	s
Anhydride		1810 and 1760	s
Acid chloride		1800	s
C-O			
Alcohols, ethers, esters, carboxylic acids, anhydrides		1300-1000	s
O-H			
Alcohols, phenols			
Free		3650-3600	m

H-bonded	3400-3200	m, broad
Carboxylic acids	3400 - 2400	m, broad
N-H		
Primary and secondary amine		
And amides		
(stretch)	3500-3100	m
(bend)	1640-1550	m-s
C-N		
Amines	1350-1000	m-s
C=N		
Imines and oximes	1690-1640	w-s
C,N triple bond		
Nitriles	2260-2240	m
X=C=Y		
Allenes, ketenes, isocyanates,		
Isothiocyanantes	2270-1940	m-s
N=O		
Nitro ($R-NO_2$)	1550 and 1350	s
S-H		
Mercaptans	2550	w
S=O		
Sulfoxides	1050	s
Sulfones, sulfonyl chlorides,		
sulfates, sulfonamides	1375-1300 and	
	1350-1140	s
C-X		
Fluoride	1400-1000	s
Chloride	785-540	s
Bromide, iodide	< 667	s

Representative ¹H Chemical shift Values (ppm)




6.0 5.9 5.8 5.7 5.6 5.5 5.4 5.3 5.2 5.1 5.0 4.9 4.8 4.7 4.6
7.5 7.4 7.3 7.2 7.1 7.0 6.9 6.8 6.7 6.6 6.5 6.4 6.3 6.2 6.1
9.0 8.9 8.8 8.7 8.6 8.5 8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6



Ph—C(=O)—H
H3C—C(=O)—H
(CH3)2CH—C(=O)—H
O2N NO2 NO2 H
10.4 10.3 10.1 10.0 9.9 9.8 9.7 9.6 9.5 9.4 9.3 9.2 9.1 9.0
OCH3
O—H
H—O—C(=O)—CH2Ph
H—O—C(=O)—CH3
H O H—O
OCH3 O H O
11.9 11.8 11.7 11.6 11.5 11.4 11.3 11.2 11.1 11.0 10.9 10.8 10.7 10.6 10.5
CH3 CH3 O H O
CH3 O H O
15.0 14.9 14.8 12.4 12.3 12.2 12.1 12.0