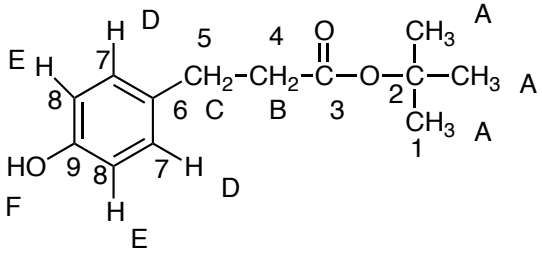
ANSWER KEY

L.I.U.

Chem. 122, Sect 009, Quiz 1, 50 pts, Spring, 2012

1. For the following molecules identify (a) the number of carbon signals (b) the number of proton signals (c) the spin-spin splittings or multiplicities of the proton signals and (d) the relative intensities of the proton signals. (10 pts)



There are 6 proton signals and 9 carbon signals.

H_A singlet, 9 H H_D doublet, 2H

H_B triplet, 2H H_E doublet, 2H

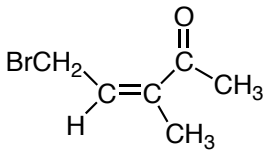
H_C triplet, 2H H_F singlet, 1H

3. In the molecule above there are several functional groups that give characteristic absorbance peaks in the infrared spectrum. Identify **three** of them and give the expected position of the absorbance peak in cm^{-1}. (6 pts)
There are several characteristic peaks in the IR spectrum. Here are a few:

O-H stretch at 3200 - 3500 cm^{-1} Carbonyl C=O stretch at 1700 - 1750 cm^{-1}

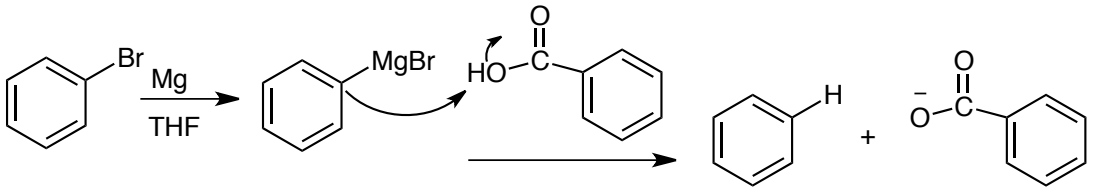
aromatic sp^2 C-H stretch at 3000 -3100 cm^{-1} sp^3 C-H stretch at 2850 - 3000 cm^{-1}

2. Identify the following molecule of formula C_6H_9BrO; IR: 1720 cm^{-1}; ^1H: δ 1.8, singlet, 3H; 2.3, singlet, 3H; 3.4, doublet, 2H, 5.6, triplet, 1H. (14 pts)



3. In the preparation of acetyl salicylic acid (aspirin) from salicylic acid, acetic anhydride and phosphoric acid (a) would water be a good recrystallization solvent for the aspirin? (b) One student transferred all her solid aspirin product to a large test tube and to save time she added 25 mL of 50:50 ethyl acetate/hexane all at once. She heated the test tube in a hot water bath. All the solid disappeared. But afterward when she cooled the test tube down in the ice bath she did not get any crystals. The teaching assistant told her to reduce the volume so she removed half of the solution with a pipette. She then cooled the test tube down again in the ice bath. She still did not get crystals. What should she do? Was her experiment ruined? How could she recover her pure aspirin? (a) Water is NOT a good recrystallization solvent since the aspirin is not soluble in water. Remember that we filtered the aspirin from water during the isolation step (b) She should add back to her test tube the solution that she removed and then reduce the total volume by evaporative heating.

4. In the preparation of triphenyl carbinol from bromobenzene, magnesium and methyl benzoate (a) What is the purpose of the flame drying? (b) Why should you first remove the stopcock from separatory funnel/addition funnel before flame drying? (c) How can you tell when you have formed your organomagnesium reagent? (d) In one lab section, the stockroom ran out of methyl benzoate ($C_6H_5CO_2CH_3$) and so the teaching assistant told the student to use benzoic acid ($C_6H_5CO_2H$) instead. Would the reaction still work? Explain briefly and show any reaction that would occur between the phenyl magnesium bromide and the benzoic acid.
(a) We first flame dry the glassware in order to remove any moisture that may be on the glassware. As we know, Grignard reagents are destroyed by water. (c) You can tell that you have formed the Grignard reagent when all or most of the magnesium chips have been consumed. (d) Benzoic acid would NOT be a good substitute for methyl benzoate, an ester, since benzoic acid has an acid proton that would be removed in a fast reaction to destroy the Grignard before it had a chance to attack the carbonyl carbon.


Br
Mg
THF
MgBr
HO
O
C
H
+
O
O
C