Name .....

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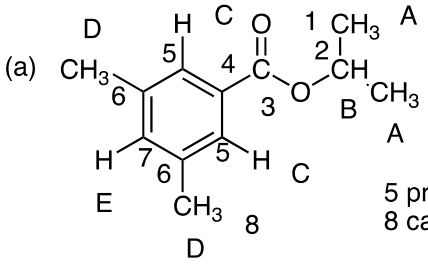
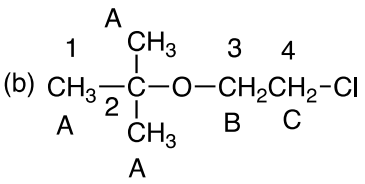
Chem. 122, Sect 012, Quiz 1, 50 pts, Spring, 2011

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. (10 pts)

(a) 

5 proton signals
8 carbon signals

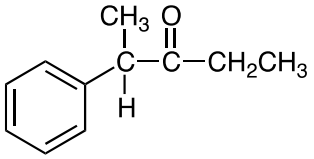
H_A doublet, 6 H H_C singlet, 2 H H_E singlet, 1 H
H_B septet, 1 H H_D singlet, 6 H

(b) 

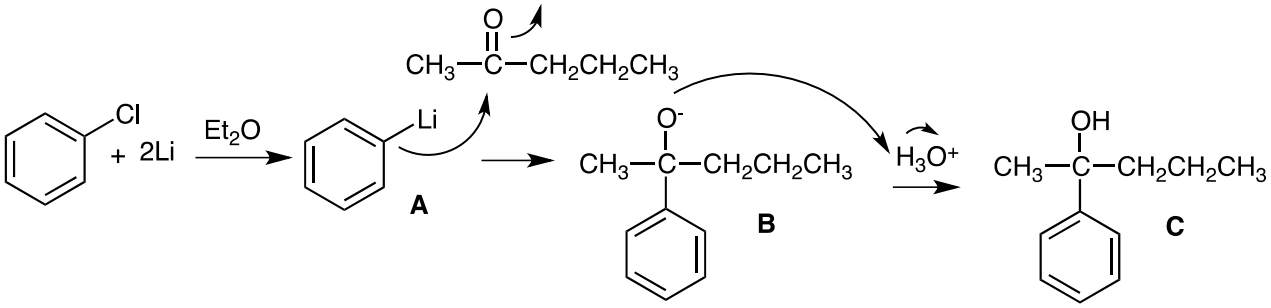
3 proton signals; 4 carbon signals

H_A singlet, 9 H ; H_B triplet, 2H; H_C triplet, 2H

2. Identify the following molecule of formula $C_{11}H_{14}O$; IR: 1750 cm^{-1}; ^1H: δ 1.2, triplet, 3H; 1.4, doublet, 3H; 1.6 quartet, 1H; 2.4, quartet, 2H; 7.5, broad singlet, 5H. (10 pts)



3. Give the product of the following reactions. (10 pts)

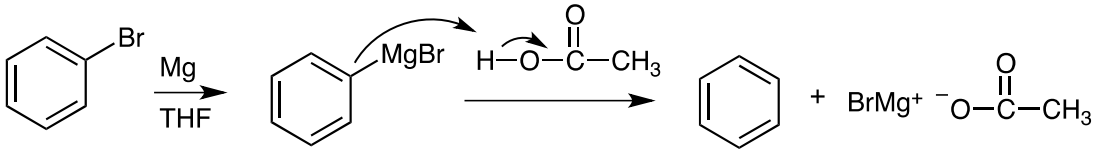


4. In the preparation of aspirin from salicylic acid [$C_4H_4(OH)(CO_2H)$*ortho*] using an excess of acetic anhydride [$(CH_3CO_2)O$] with a catalytic amount of phosphoric acid, we heated the reaction mixutre for 5 minutes in a water bath. Then we added 2 mL of water, we reheat for 1-2 minutes and then we added 20 mL of cold water. (a) What is the purpose of adding the 2 mL of water first? (b) What is the purpose of then adding the 20 mL of cold water? (c) How did we collect the product? (d) On doing the recrystallization from ethyl acetate and hexane, some people noticed a small, clear layer on the bottom of their test tube after all the aspirin had dissolved. What was this layer and how could you get rid of it? (12 pts)

ANS: (a) to hydrolyze excess acetic anhydride (b) the 20 mL cold water was to precipitate the product; aspirin is not soluble in cold water (c) We collected the product by vacuum filtration. (d) This was water due to not getting the crystals sufficiently dry during the vacuum filtration

5. In the preparation of triphenylcarbinol from bromobenzene, magnesium and methyl benzoate ($C_6H_5CO_2CH_3$) in THF, the stockroom ran out of THF. The person working in the stockroom (an undergraduate work-study student) told one student to use acetic acid (CH_3CO_2H) instead. (a) Was this a good choice of solvents? Explain and show any reactions that may have occurred. (b) What would be a better substitute for THF as a solvent? (i) ethanol (ii) 1,4-dioxane (iii) acetone. Explain briefly, discussing each of the choices in turn. (8 pts)

Ans: (a) No, the Grignard would react with the acetic acid as follows:



(b) 1,4-dioxane would be best since it is an ether, like THF, and has a similar polarity. Ethanol has an acidic proton and would destroy the Grignard, like acetic acid. Acetone has a ketone and would react with the Grignard.