

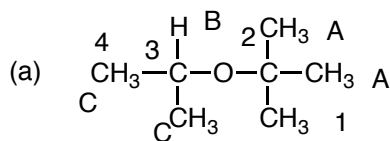
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Answer Key L.I.U.

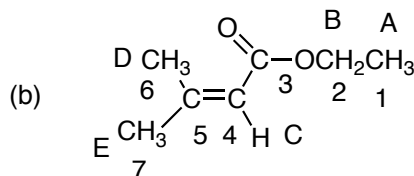
Chem. 122, Sect 007,

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)

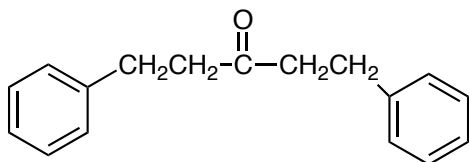


There are three proton signal and four carbon signals. In the tertiary butyl group, H<sub>A</sub>, all 9 protons are the same due to free rotation and give a singlet (no neighbors). The 6 H<sub>C</sub> protons are also all the same; they are split into a doublet by H<sub>B</sub> and H<sub>B</sub> is a septet, due to its 6 neighbors.

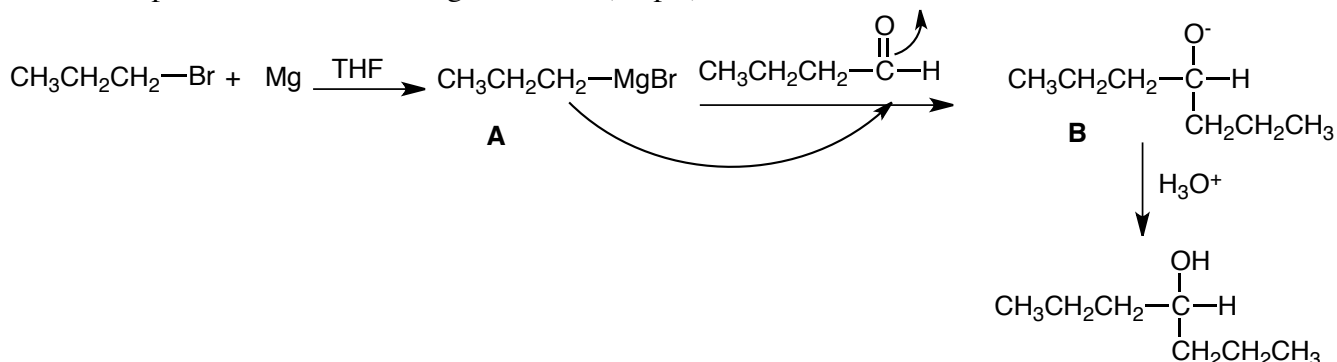


H<sub>A</sub> is a triplet, H<sub>B</sub> is a quartet, H<sub>C</sub>, H<sub>D</sub> and H<sub>E</sub> are all different since there is no free rotation around the C=C bond and are all singlets. Each carbon is in a different environment so there are 7 carbon signals.

2. Identify the following molecule of formula C<sub>17</sub>H<sub>18</sub>O; IR: 1740 cm<sup>-1</sup>; <sup>1</sup>H: δ 2.2, triplet, 4H; 2.3, triplet, 4H; 7.2-7.5, broad singlet, 10 H. (10 pts)



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




4. In the recrystallization of aspirin from hexane and ethyl acetate, the general procedure is to transfer all of the solid to a medium test tube, cover the solid with ethyl acetate and hexane and heat the test tube to boiling in a water bath. (a) What should you do if the solid does not dissolve completely when the solvent mixture starts to boil? (b) What is the purpose of keeping the spatula in the test tube? (c) What would be the best substitute for ethyl acetate as a co-solvent with hexane? (i) water (ii) propane (iii) acetone (CH<sub>3</sub>COCH<sub>3</sub>). Explain briefly. (10 pts)

ANSWERS: (a) If the solid does not dissolve completely when the solvent starts to boil, add more solvent quickly, bring the new mixture to boil and if it is still not dissolved add more solvent in small amounts until all the solid dissolves. Do not leave the test tube in the boiling water bath since the solvent will simply boil away. Work quickly when doing the recrystallization. (b) The spatula is for stirring your mixture but more importantly it acts as a boiling chip. (c) The best substitute is acetone. Water is not miscible with hexane and propane is a gas AND it is non-polar.

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5. In the preparation of triphenylcarbinol from bromobenzene, magnesium and methyl benzoate ( $C_6H_5CO_2CH_3$ ) in THF (a) What was the purpose of flame drying the glassware? (b) What would be a good substitute for THF as a solvent? (i) ethanol (ii) 1,4-dioxane  (iii) hexane. Explain briefly, discussing each of the choices in turn. (c) Why was it important to have the pH of the reaction mixture be acidic before doing the steam distillation? Explain briefly and show any reactions that occurred on adding the aqueous acid. (10 pts)

Dioxane would be the best substitute for THF. It is an ether like THF and has similar polarity. Ethanol would not be good because it has an acidic proton and would destroy the Grignard as soon as it forms and hexane would not be good because the Grignard reagent is very polar and not very soluble in the non-polar hexane.