

Lab Report:

Synthesis of an Ant Alarm Pheromone:

2-Methyl-2-Heptanone

NAME: _____

PARTNER'S NAME: _____

LAB SECTION: _____

DATE: _____

% SCORE:

	Pts possible	Pts received
Lab Notebook		
Abstract		
Spectroscopy & Mechanism		
Questions		
Discussion		
Total		

A. ABSTRACT: Be brief but summarize the two-week lab. Include % yield from each part and overall yield in your abstract.

B. MECHANISM AND SPECTROSCOPIC ANALYSIS

1. REACTION MECHANISM

- a. In formation of the Grignard reagent Mg is converted to magnesium with a plus two oxidation state. Circle the reaction type magnesium is undergoing.

Oxidation	Reduction	Nucleophilic Substitution	
Electrophilic Substitution		Electrophilic Addition	Elimination

- b. What reaction type is 1-chloro-2-methylpropane undergoing?

Oxidation	Reduction	Nucleophilic Substitution	
Electrophilic Substitution		Electrophilic Addition	Elimination

- c. What reaction type is butanal undergoing in its initial reaction with the Grignard reagent?

Oxidation	Reduction	Nucleophilic Substitution	
Electrophilic Substitution		Electrophilic Addition	Elimination

2. IR SPECTROSCOPY

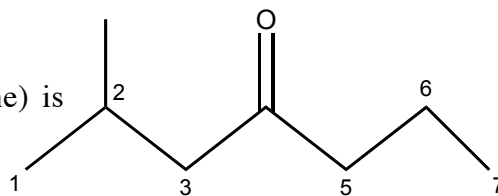
a. Attach your alcohol spectrum. Label the spectrum with your name, the sample and assign the peaks consistent with bond vibrations of 2-methyl-4-heptanol.

b. Attach your ketone spectrum. Label the spectrum with your name, the sample and assign the peaks consistent with bond vibrations of 2-methyl-4-heptanone.

2. ¹H NMR SPECTROSCOPY

¹H NMR spectrum of the ketone (2-methyl-4-heptanone) is provided.

a. Report the chemical shift of each proton signal as labeled on the structure shown. Briefly state how you assigned the peak using splitting, integration and molecular structure.



H1

H2

H3

H5

H6

H7

b. Show the calculation of the coupling constants between protons 3 and 2, J_{32} and protons 5 and 6, J_{56}

J_{32}

J_{56}

C. QUESTIONS

1. There are 8 peaks in the carbon NMR spectrum of the alcohol. What does this imply about the relationship between the two methyl groups that appear identical? Are they homotopic, enantiotopic or diastereotopic? Draw structures to help illustrate your answer.
2. In the workup of Grignard reaction the quenched reaction mixture is poured into a separatory funnel and two layers exist. Note which layer, organic or aqueous, you would expect each compound to be present in the higher concentration:
 - a. 2-methyl-4-heptanol _____
 - b. MgCl_2 _____
 - c. Unreacted butanal _____
 - d. Unreacted 1-chloro-2-methylpropane _____
 - e. Excess HCl _____
3. In the extraction process to isolate the alcohol product you washed the ether layer with saturated sodium chloride. What role does the saturated sodium chloride wash play? Explain
4. In the reaction with bleach to form a ketone the alcohol is oxidized. What reagent is the oxidizing agent? Which element in the reagent is acting as the oxidizing agent? How many electrons does the alcohol lose as it is oxidized to a ketone?
5. Calculate the yield of ketone in grams in the oxidation reaction using your GC results.
6. Calculate the overall percent yield of ketone from 1-chloro-2methylpropane in your 2-step reaction sequence of alcohol formation and oxidation.

7. Why does the ketone have a shorter retention time than the alcohol in GC?
8. The ionized ketone undergoes McLafferty rearrangements in the MS. Show the mechanism for one of the McLafferty rearrangements and cite m/z of the products formed.
9. The MS trace of the alcohol has no parent ion. Can you offer an explanation for this?
10. The alcohol oxidation with bleach can be considered “green chemistry” because acetic acid solvent and chloride by product are non-toxic. What other reagent could be used to oxidize an alcohol to a ketone?

D. DISCUSSION

Briefly discuss your two-week synthesis of the ant pheromone. Was your synthesis successful? Support your answer with data you collected – specifically- cite data which identify the alcohol and ketone. If you did not obtain product or had greater than 100% yield of a step offer an explanation. Many alarm pheromones are lower molecular weight compounds, why do you think this is true?