

Lab Report: Synthesis 2, 2-Dimethyl-1, 3-Dioxolane

NAME: _____

PARTNER'S NAME: _____

LAB SECTION: _____

DATE: _____

% SCORE:

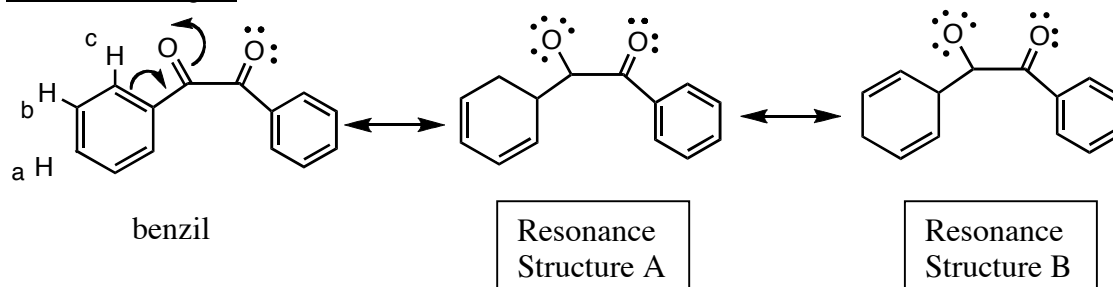
	Points possible	Points received
Lab Notebook		
Abstract		
NMR		
Questions		
Conclusions		
Total		

A. ABSTRACT:

B. ¹H NMR ANALYSIS

1. HNMR of Benzil

a. Follow the flow of electrons shown in benzil and complete **Resonance Structure A** by adding bonds and charges. Then show flow of electrons from A to B and complete **Resonance Structure B** adding bonds and charges.



b. Assign the chemical shifts to the labeled protons in benzil by writing the chemical shift (ppm) next to the appropriate proton. Briefly explain how you assigned each signal using splitting and the resonance structures. The H-NMR of benzil has been reported as follows:

7.7 ppm (d, 4H), 7.55 ppm (t, 2H), 7.43 ppm (t, 4H)

Ha

Hb

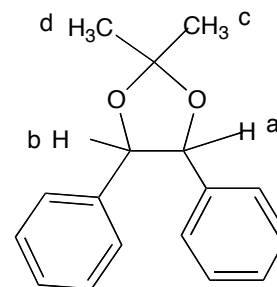
Hc

2. HNMR SPECTRA OF THE 2,2-DIMETHYL-1,3-DIOXOLANE PRODUCT

Label the provided HNMR spectrum of the acetal product with the letters on the structure below. Attach the labeled HNMR to your report. Don't worry about which peak you assign to each methyl group, they are not possible to distinguish with your information

a. According to the HNMR provided is the relationship between the protons a and b diastereotopic? Explain

b. Are protons c and d on the methyl groups diastereotopic? Explain

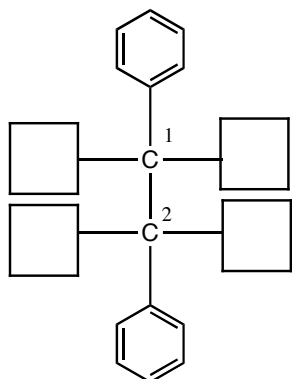


c. Could you use this HNMR to determine if protons were enantiotopic or homotopic?

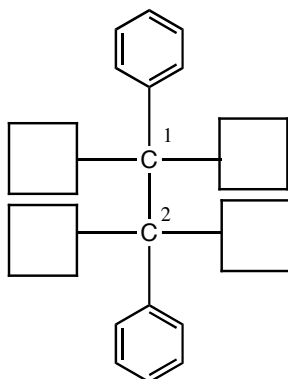
C. QUESTIONS

1. Calculate the theoretical weight of sodium borohydride needed to completely reduce 350mg of benzil. Show your calculations (hint: consider the mole ratio of sodium borohydride to benzil)
2. The Jones test is used to detect a 1° or 2° hydroxyl group, Offer an explanation for why the test does not identify 3° hydroxyl groups.

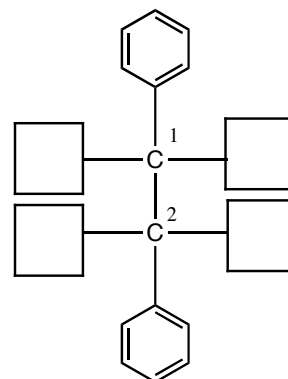
3. Complete the Fischer projections of the hydrobenzoin isomers with H and OH to agree with the assigned configurations.



1R, 2R

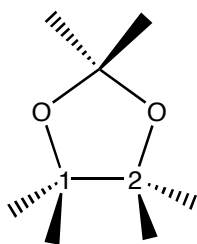


1S, 2S

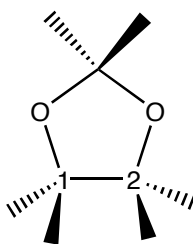


1R, 2S

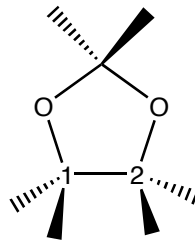
4. Which structures above will have identical HNMR spectra? _____
5. Explain the role of the iron (III) chloride in the formation of the acetal.
6. Complete each of the structures below with hydrogen, phenyl and methyl groups to make the assigned isomers of the dioxolane (acetal) product.



(1R,2R)



(1S,2S)



(1R,2S)-meso

7. Considering the symmetry of the above structures, predict the number of methyl peaks that will be observed in the ^1H -NMR spectrum of the each dioxolane.

1S,2S ____ 1R,2R ____ 1R,2S ____

D. CONCLUSIONS

1. Based on your melting point what is the stereochemistry of the hydrobenzoin you formed in lab: meso (1R,2S) or racemic (1R,2R and 1S,2S)? _____
2. Based upon the HNMR of your acetal product (analyzed in section B 2) and the symmetry of the acetal methyl groups (question 6 & 7) what is the stereochemistry of the acetal formed in lab: meso (1R,2S) or racemic (1R,2R and 1S,2S)? _____
3. Should the stereochemistry of hydrobenzoin's benzylic carbons change when it forms the acetal? Explain
4. Does the sodium borohydride reduction of benzil follow Cram's Rule for addition to carbonyls? Use structures to illustrate your answer.