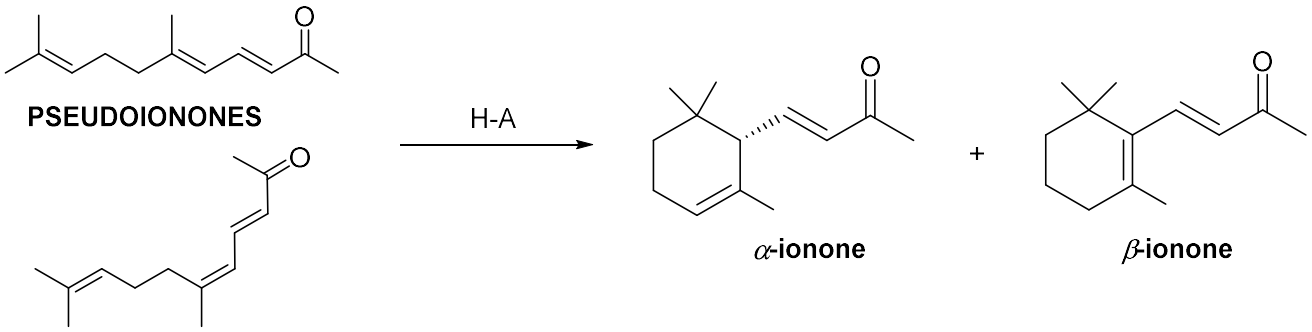
CHEM 110L, Lecture 4

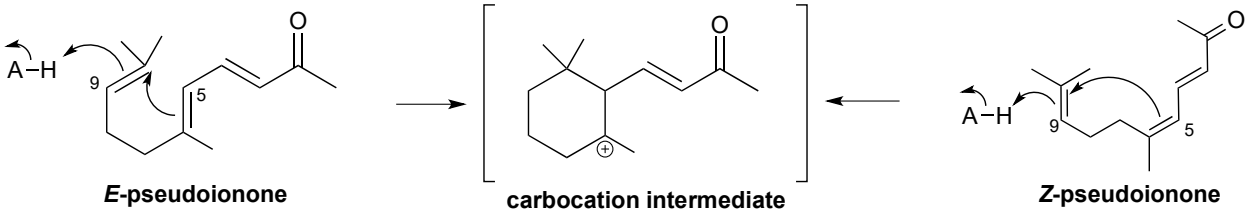
Experiment 3, Week 2 – Ionone Synthesis: Acid-Catalyzed Cyclization of Pseudoionones,

Reactions Overview

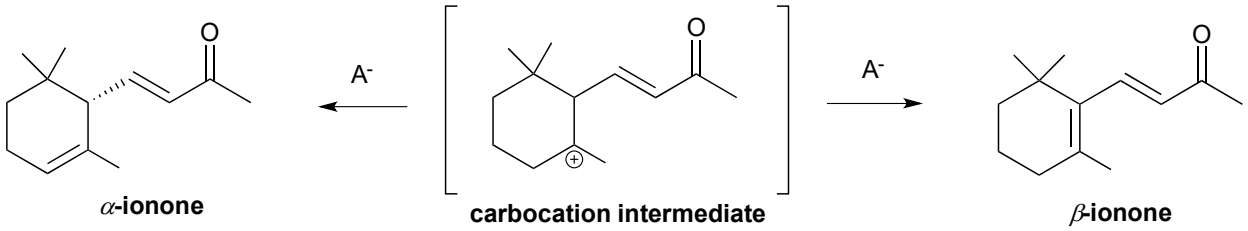
H-A options: H_2SO_4 / HAc or H_3PO_4



Intramolecular Alkene Addition



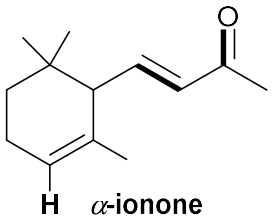
Alkene Formation (E1-Style)



Sulfuric / Acetic Acid Rxn Workup: Prepare a mixture of 30 mL of cold water and 6 mL of BME in a flask. Swirl, then transfer to the reaction mixture, mix, and transfer it to a separatory funnel. Extract the product into the organic layer. Separate the layers and extract the aqueous layer with an additional 6 mL of BME. Wash the combined organic layers with 2 x 12 mL of an aqueous solution containing $NaHCO_3$ (5% w/v) and NaCl (10% w/v).

Phosphoric Acid Rxn Workup: Add 30 mL of aqueous NaCl (10% w/v) and transfer the mixture into a separatory funnel. Wash the flask with 15 mL of BME and transfer the wash to the separatory funnel. Mix and separate the layers. Extract the aqueous layer again with 15 mL of BME. Wash the combined organic layers first with 15 mL of an aqueous solution containing $NaHCO_3$ (5% w/v) and NaCl (10% w/v), followed by 15 mL of aqueous NaCl.

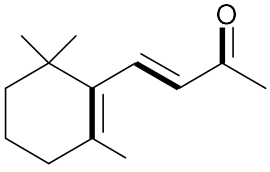
IR & UV-vis Spectroscopy of Products



α-ionone

Conjugated system with 3π orbitals

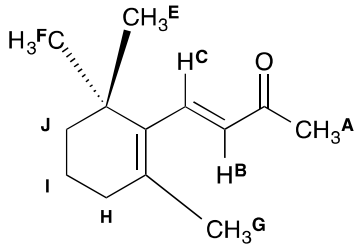
Additional vinylic C-H bend: IR 600-900cm^{-1}



β-ionone

Conjugated system with 3π orbitals

Strong UV absorbance at 295nm



β-ionone

^{1}H NMR Analysis of β-ionone

Signal	Integration (#H's)	Splitting (exp/obs)	Chemical Shift, Expected	Chemical Shift, Observed (Fig 20.3)
A	3			
B	1			
C	1			
D	-- N/A --			
E	3			
F	3			
G	3			
H	2			
I	2			
J	2			

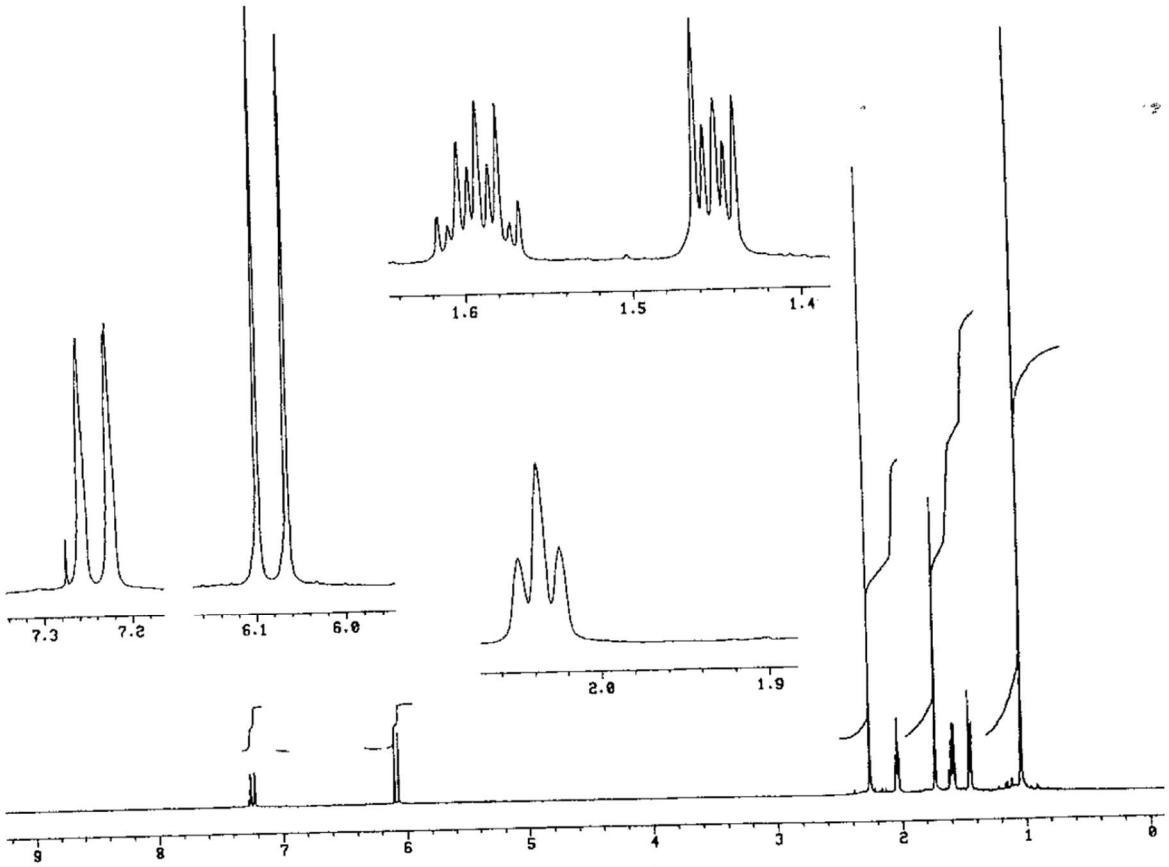
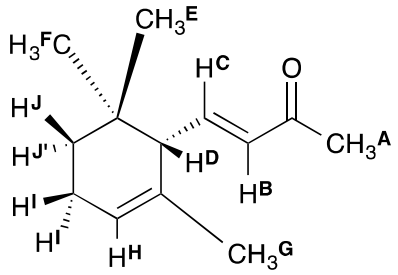


Figure 23.15 500-MHz ^{1}H-NMR spectrum of β-ionone in $CDCl_3$.



α-ionone

^{1}H NMR Analysis of α-ionone

Signal	Integration (#H's)	Splitting (exp/obs)	Chemical Shift, Expected	Chemical Shift, Observed (Fig 20.3)
A	3			
B	1			
C	1			
D	1			
E	3			
F	3			
G	3			
H	1			
I	2			
J	1			
J'	1			

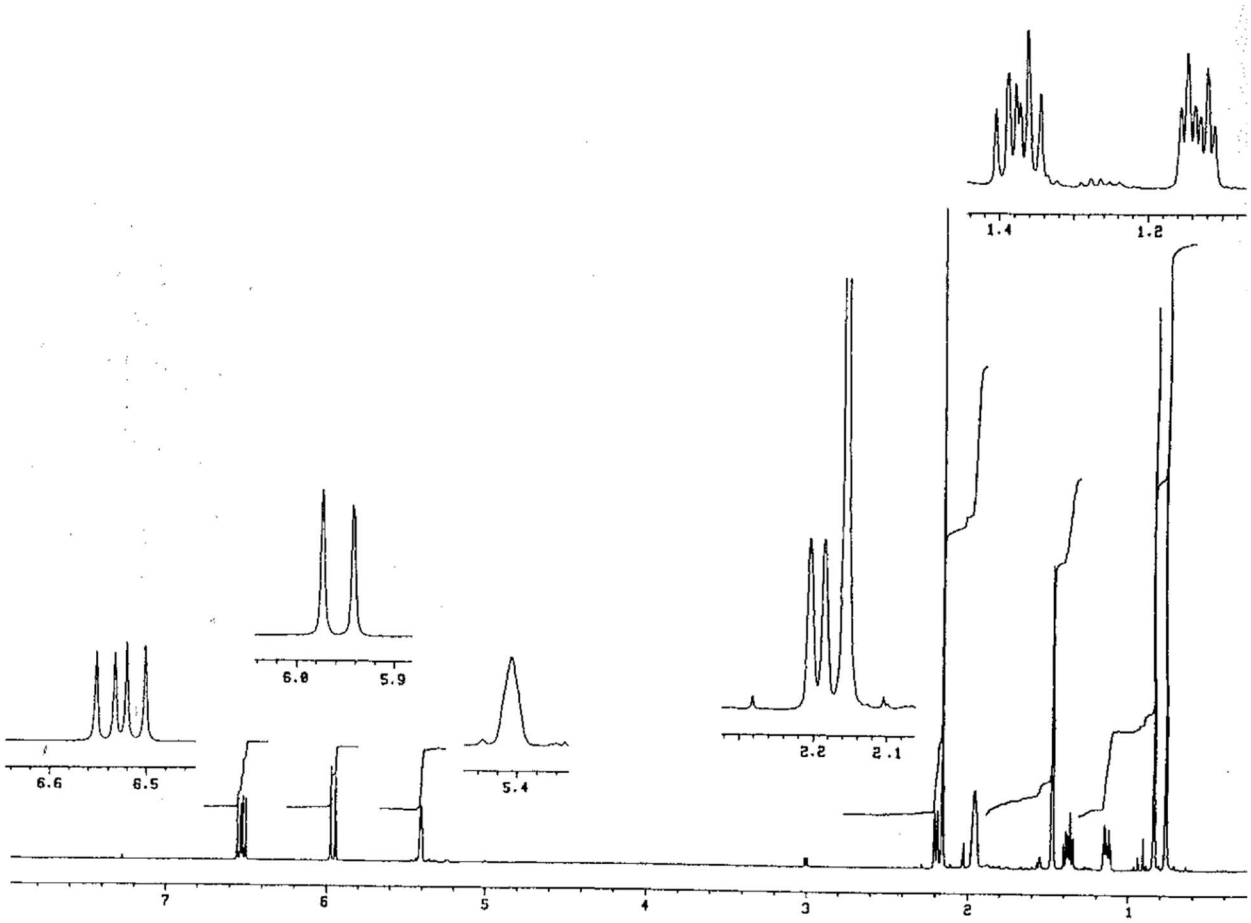


Figure 23.12 500-MHz ^{1}H-NMR spectrum of α-ionone in $CDCl_3$.