Name _____ Partner _____

TA Name _____ Section Letter _____ Day _____ Time _____

Experiment 5B Worksheet – Pseudoionone Cyclization – Ionone Synthesis

Use as reference for notebook preparation – submit on Canvas this individually after lab

A. Experimental Purpose and Ionone Synthesis Reaction Scheme

B. Reagent Table – choose one cyclization procedure to fill out this table

Refer to the procedure for amounts and safety table for hazards; find the chemical properties on Wikipedia!

Name	Volume	Density	Mass	MW	mmol	Equiv*	Boiling / melting point	Hazards
pseudodionones (crude product)	-							
		-	-	-				
					-	-		
		-	-	-	-	-		
ionones (crude product)	-	-				-		

*** Equiv** = molar equivalents of reaction components with respect to the limiting reagent (pseudoionones)

- reagent equivalents: divide the mmol of reagent by the mmol of pseudoionones

C. Procedure Diagrams - on as many pages as needed

- All **labeled equipment, chemical names** with **amounts**, **transfers**, **cleanup & safety** notes
  - Help w diagrams: Slugs@home Exp 5 website & class notes

1. **Reaction setup** – all equipment and chemicals (name, structure, and amount)

2. **Reaction workup** – flow chart / diagrams of separatory funnel contents of layers, all solution transfers, and rota-vap

3. **Analysis** – GC, IR, and UV-vis sample preparation; sketches of spectra, identifying key signals

D. Partner Agreement / Accountabilibuddy Contract: Both students in the pair get the same lab report grade. There is also the option to submit individual reports – please do what works best for you and your partner. Split up partner assignments in part **(a)** and schedule a time to collaborate after lab in part **(b).**

(a) *Didn't we do this last week?* Yas and it's great to check-in with your partner 😊 Students are encouraged to work on report together during lab. The assignments below indicate who will put together or type the **final responses**.

Name		
Abstract		
In-Lab Questions		

(b) "DO" Date: _____ = when / how you'll meet or exchange work to discuss & proofread, at least 1-2 days before the DUE date

E. Data & Analysis

Mass of pseudoionones _____ mg Theoretical yield of ionones _____ mg

Theoretical Yield Calculation:

Miscellaneous notes & observations – ex. Suspected sources of product loss

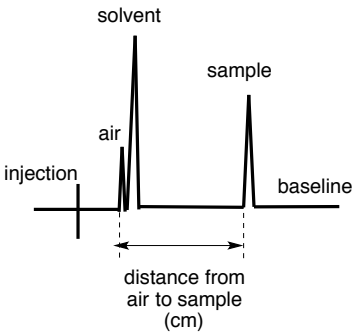
Empty RBF mass _____ g After rota-vap: mass of RBF & crude product _____g

Crude product mass (actual yield) _____g

Percent Yield = [(actual yield) / (theoretical yield)] x 100% _____% yield, ionones

GC Analysis of Product Mixture – Ionones

Peak #	Peak ID**	Corrected t_R (s)	Integration (cm^2)	% Composition



$$t_R' \text{ (sec)} = \text{distance from air to sample (cm)} \times \frac{\text{chart speed } \mathbf{1 \text{ min}}}{\mathbf{2.5 \text{ cm}}} \times \frac{\mathbf{60 \text{ sec}}}{\mathbf{1 \text{ min}}}$$

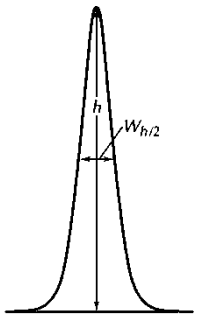


FIGURE 20.13
Determining peak area: h = height; $W_{h/2}$ = width at half-height.

Integration: Peak Area = h x $w_{h/2}$

Crude product mixture - ionones IR – draw structures

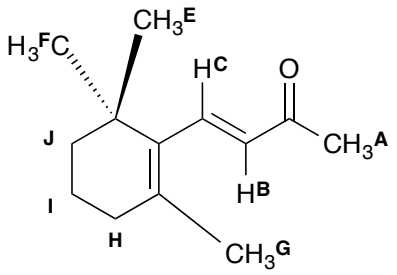
Functional Group	Bond	Expected Wavenumber Range (cm^{-1})	Observed Wavenumber (cm^{-1})

Pure Spectra Provided - alpha-ionone IR – draw structure

Functional Group	Bond	Expected Wavenumber Range (cm^{-1})	Observed Wavenumber (cm^{-1})

Pure Spectra Provided - beta-ionone IR – draw structure

Functional Group	Bond	Expected Wavenumber Range (cm^{-1})	Observed Wavenumber (cm^{-1})



^{1}H NMR Analysis of β-ionone **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			

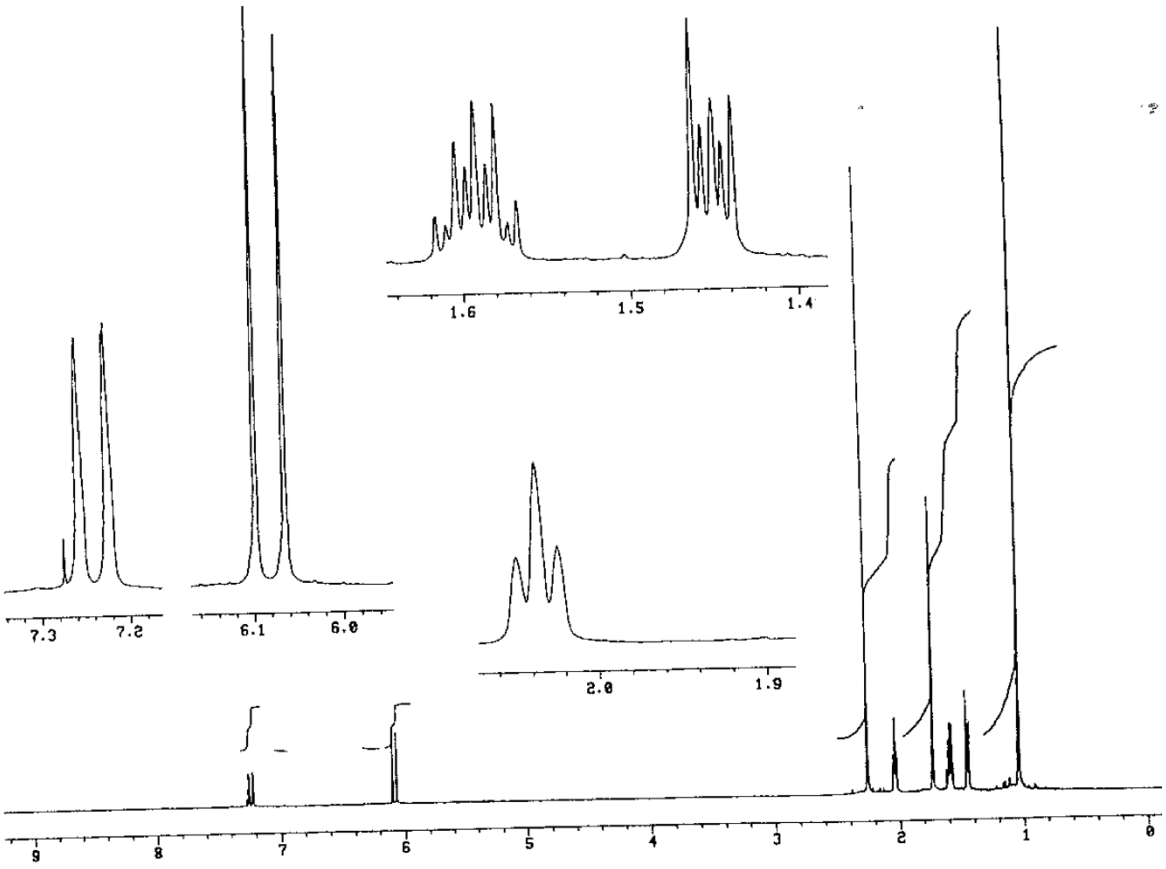
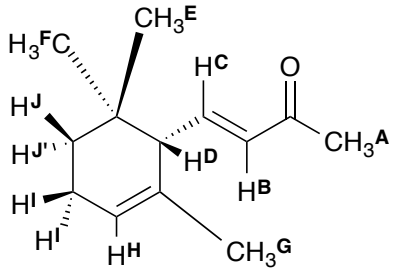


Figure 23.15 500-MHz ^{1}H-NMR spectrum of β-ionone in $CDCl_3$.



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			

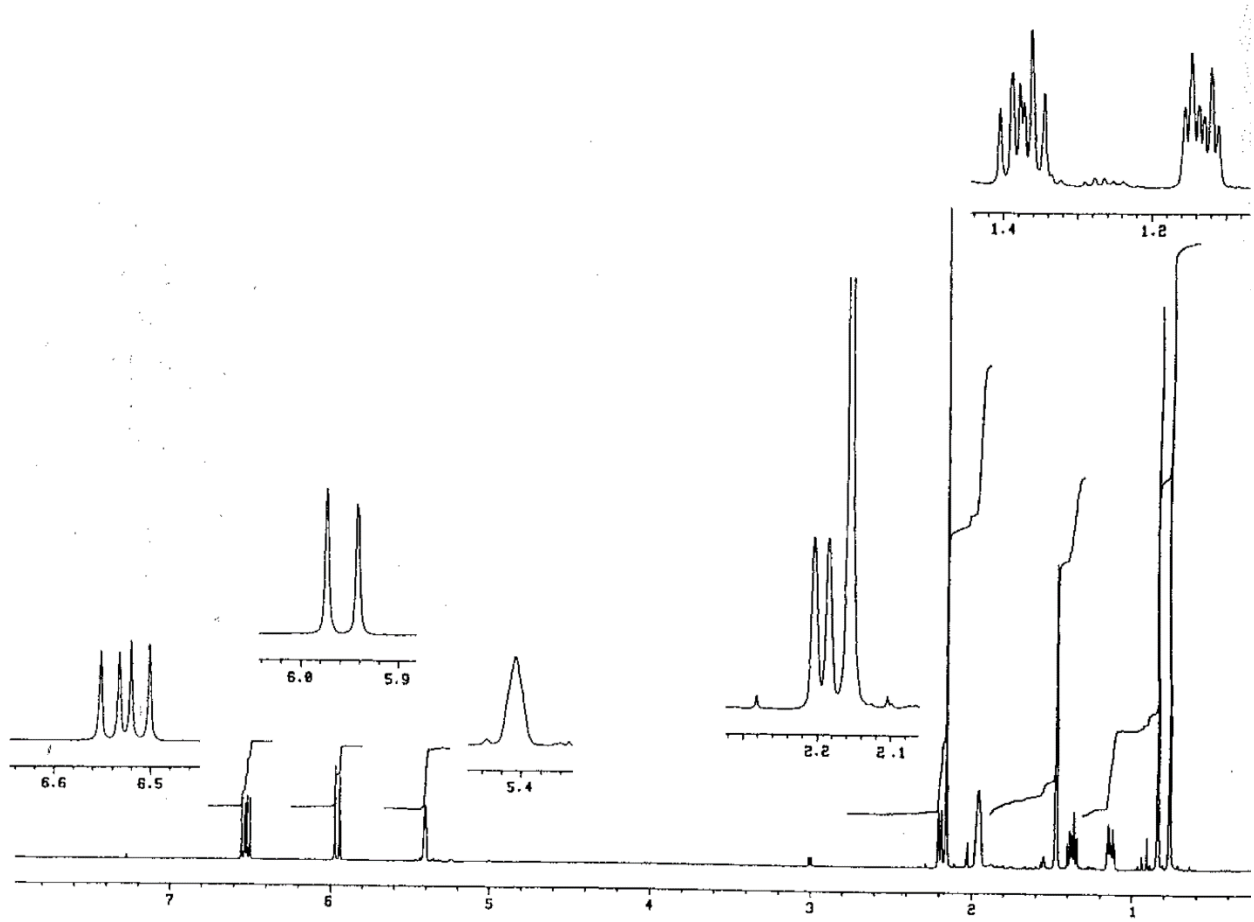


Figure 23.12 500-MHz ^{1}H-NMR spectrum of α-ionone in $CDCl_3$.