Name _____ Partner _____

TA Name _____ Section Letter _____ Day _____ Time _____

Experiment 5A Worksheet – Pseudoionone Synthesis from Citrals

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

A. Experimental Purpose and Pseudoionone Synthesis Reaction Scheme

B. Reagent 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
Citrals (1:1 mixture)	-							
Acetone								
2.25 M NaOEt Sodium ethoxide in ethanol		-	-	-				
2 M HCl								
Tert-butyl methyl ether (BME)					-	-		
10% NaCl *(aq)*		-	-	-	-	-		
pseudodionones (crude product)	-	-				-		

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

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

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 and IR 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) *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 citrals _____ mg Theoretical yield of pseudoionones _____ 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, Pseudoionones

Citrals IR

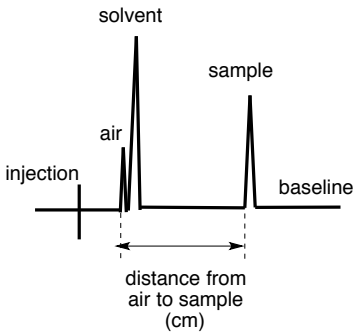
Functional Group	Bond	Expected Wavenumber Range (cm^{-1})	Observed Wavenumber (cm^{-1})

Pseudoionones IR – draw structures

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

Gas Chromatography (GC) Analysis – week 1 and/or 2

Standard Sample	Corrected t_R' (s)
citrals peak 1	
citrals peak 2	
Pseudoionone peak 1	
Pseudoionone peak 2	
a-Ionone	
b-Ionone	



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

Gas Chromatography (GC) – Crude Psuedoionones Product

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

** Use corrected retention times to assign each peak to one of the standards.
Note that not all standards may be present, some peaks overlap, and other unknown peaks may appear.