

Name _____

TA Name _____

Section Letter _____ Day _____ Time _____

Experiment 3AB Worksheet – Synthesis of Phenacetin

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

Pre-Lab Requirements

1. **Dress for lab** – see safety rules – arrive a few minutes early
2. Copy these templates into your lab notebook – contact instructors for alternate accommodations
 - Fill in the **purpose with structures and reagent table**
 - **Procedure Diagrams** – must be complete before you can start the lab

A. Experimental Purpose and Phenacetin 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 or melting point	Hazards
Acetaminophen (ACE)	-	-						
Potassium carbonate (K ₂ CO ₃)	-	-						
Acetonitrile (CH ₃ CN)					-			
Ethyl iodide (Etl)								
Tert-butyl methyl ether (BME)					-	-		
5% NaOH		-	-	-	-	-		
sat. NaCl		-	-	-	-	-		
phenacetin (product)	-	-						

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

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

- solvent equivalents = approximate concentration: divide the mmol of ACE by mL of acetonitrile

D. Data & Analysis

Exp 3B. Phenacetin Analysis

Thin-Layer Chromatography (TLC)

- Sketch each plate
- Label each lane / spot
- Calculate R_f values for each spot

Melting Temperature

	Melting Starts...	Melting Ends...
Acetaminophen		
Product		

Phenacetin IR

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

D. Data & Analysis

Exp 3B. Phenacetin Analysis

Ferric Chloride Test for Phenols

Sample	Observation	Interpretation
Acetaminophen		
Product (phenacetin)		
Water		

¹H NMR of acetaminophen
(draw structures with labels)

¹³C NMR of acetaminophen

Signal	Integration (#H's)	Splitting	Chemical Shift Expected (ppm)	Chemical Shift Observed (ppm)	Signal	Chem Shift Expected (ppm)	Chem Shift Observed (ppm)
A					A'		
B					B'		
C					C'		
D					D'		
E					E'		
					F'		

D. Data & Analysis

^1H NMR of phenacetin
(draw structure with labels)

^{13}C NMR of phenacetin

Signal	Integration (#H's)	Splitting	Chemical Shift Expected (ppm)	Chemical Shift Observed (ppm)	Signal	Chem Shift Expected (ppm)	Chem Shift Observed (ppm)
A					A'		
B					B'		
C					C'		
D					D'		
E					E'		
					F'		