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!

			•				Boiling or melting	Hazards
Name	Volume	Density	Mass	MW	mmol	Equiv*	point	i iazai us
Acetaminophen (ACE)	-	-						
Potassium carbonate (K ₂ CO ₃)	-	-						
Acetonitrile (CH₃CN)					ı			
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

C. Procedure Diagrams - on a	as many pages as needed
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- All labeled equipment, chemical names with amounts, transfers, cleanup & safety notes
 - o Help w diagrams: Slugs@home Exp 3 website & class notes
- **1. Reaction setup** all equipment and chemicals (name, structure, and amount)
- 2. Reaction workup flow chart / diagrams of screw-cap test tube contents and all solution transfers
- **3. Analysis** Ferric chloride test procedure; NMR and IR sample preparation; sketches of spectra, identifying key signals

	Functional Group	Bond	Expected Range (c	Wavenumber m ⁻¹)	Observed Wa (cm ⁻¹)	venumber	
Aceta	ıminophen IR						
Perce	nt Yield = [(actual yield	l) / (theoret	ical yield)] :	x 100%	%	Yield of phena	cetin
Yield	of phenacetin =	n	ng				
	Product Loss notes:						
	Theoretical Yield Cald	culation:					
Mass	of acetaminophen		mg	Theoretical yield	of phenacetin _	mg	
Exp 3	A. Phenacetin Synthe	esis					

Exp 3B. Phenacetin Analysis

Thin-Layer Chromatography (TLC)

- Sketch each plateLabel each lane / spot
- Calculate R_f values for each spot

Melting Temperature

morang romporatoro			
	Melting Starts	Melting Ends	
Acetaminophen			
Product			

Phenacetin IR

Functional Group	Bond	Expected Wavenumber Range (cm ⁻¹)	Observed Wavenumber (cm ⁻¹)

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)
Α					A'		
В					B'		
С					C'		
D					D'		
E					E'		
					F'		

¹H NMR of phenacetin (draw structure with labels)

¹³C 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)
Α					A'		
В					B'		
С					C'		
D					D'		
E					E'		
					F'		