Name _____

TA Name _____ Section Letter _____ Day _____ Time _____

Experiment 3C Worksheet – Bromination 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 Bromination 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
Phenacetin	-	-						
potassium bromate								
Acetic acid, glacial								
HBr $_{(aq)}$ 48% w/w								
sodium thiosulfate (1 M)					-	-		
Water					-	-		
bromophenacetin (product)	-	-						

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

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

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 3 website & class notes

1. **Reaction setup** – all equipment and chemicals (name, structure, and amount)
2. **Reaction workup** – flow chart / diagrams of container contents and all solution transfers
3. **Recrystallization**
4. **Analysis** –same as Part B for NMR and IR analysis

D. Data & Analysis

Exp 3C. Phenacetin Bromination

Mass of phenacetin _____ mg Theoretical yield of bromophenacetin _____ mg

Theoretical Yield Calculation:

Product Loss notes

Crude yield of bromophenacetin = _____ mg

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

_____% Yield of bromophenacetin

Bromophenacetin IR

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

D. Data & Analysis

Melting Temperature

	Melting Starts…	Melting Ends…
Acetaminophen		
Phenacetin		
Bromophenacetin product		

^{1}H NMR of bromophenacetin
(draw structure with labels)

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

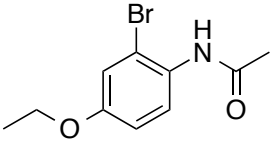
Exp 4C. Recrystallization

Crude yield _____mg Recrystallized recovery _____mg

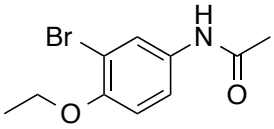
% Recovery = [(recrystallized mass) / (crude yield)] x 100%

_____ % Recovery from recrystallization

Which product did you make?!



N-acetyl-2-bromo-4-ethoxyaniline
mp 96-97 °C



N-acetyl-3-bromo-4-ethoxyaniline
mp 112-114 °C