

Name _____

Partner Name _____

TA Name _____

Section Letter _____ Day _____ Time _____

Experiment 5 Worksheet – Synthesis of Aspirin

Use as reference for notebook preparation – everyone submits on Canvas individually after lab

Pre-Lab Requirements

1. **Dress for lab** – see safety rules – arrive a few minutes early
2. **Lab Notebook:** copy templates below into designated notebook
 - **Purpose, scheme, and reagent table**
 - **Procedure Diagrams** – must be complete before you can start the lab

A. Experimental Purpose and 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
Salicylic acid	-							
Acetic anhydride		-						
Phosphoric acid	2 drops	-	-		-			
Acetylsalicylic acid (aspirin, product)	-							

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

- Acetic Anhydride (reagent): divide the mmol of reagent by the mmol of salicylic acid

See Slugs@home for pics & videos of the full lab!

C. Procedure Diagrams - use many pages as needed, at least 3 is typical

- Use the procedure in the lab PDF to create your hand-drawn experimental instructions
 - Simple sketches & labels for **all equipment, chemical names with amounts, & transfers**
- Format: Break it up with flow charts, bullet-points, comic strip, and/or whatever works for you!
 - Avoid copying the procedure word-for-word.
 - Make it easy for anyone to follow your procedure without referring to this document.
- **Slugs@home Exp 4 website** - Equipment & Safety pages; pictures & videos of the whole lab
- The **class notes** include useful diagrams as well

1. **Reaction Set-up** – chemicals added to flask, assembly of reaction apparatus

2. **Reaction Work-Up** – acidification and crystallization

3. **Filtration** - isolation of aspirin from solution

4. **Analysis: Iron (III) Chloride tests and IR Spectroscopy** – labeled test tubes with contents and observations; preparation of IR sample and sketch of IR spectrum

D. Accountability Buddy Contract: you have the OPTION to work with one PARTNER to submit one report and get the same grade in GradeScope. Add your name to one box in part **(a)** and schedule a time to collaborate after lab in part **(b)**. If you prefer to work on INDIVIDUALLY on this report, please include that in your notebook page submission.

(a) Who's finalizing what? Discuss the in-lab questions in the Exp 5 PDF with your partner during / after lab. Use the writing worksheet toward the end of this document for step-wise instructions on writing the experimental methods 😊 Decide who will type or draw the revised responses to which in-lab questions.

In-Lab Q's # _____ / Exp Methods	In-Lab Q's # _____ / Exp Methods

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

Who will combine both sets of in-lab questions and submit as one PDF to GradeScope? _____

E. Data

Salicylic acid mass _____ mg

Theoretical Yield _____ mg

Calculation:

Product Loss

Product Recovery _____ mg

% Yield _____

E. Data (cont'd)**Ferric Chloride Test Results**

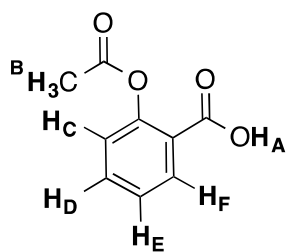
Sample	Observations & brief interpretation
Salicylic Acid	
Product	
Water	

IR Spectrum of Salicylic Acid

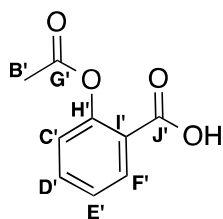
Functional Group	Bond Assignment (C=O, N-H, etc.)	Expected Wavenumber Range (cm⁻¹)	Wavenumber (cm⁻¹)

IR Spectrum of Aspirin

Functional Group	Bond Assignment (C=O, N-H, etc.)	Expected Wavenumber Range (cm⁻¹)	Wavenumber (cm⁻¹)

E. Data (cont'd)**¹H NMR Analysis of Aspirin** – posted on Canvas**Acetylsalicylic Acid (Aspirin)**

Signal	Integration	Splitting	Expected Chemical Shift (ppm)	Observed Chemical Shift (ppm)
A	1 H	singlet	9.7 – 12.5	10
B				
C				
D				
E				
F				

¹³C NMR Analysis of Aspirin – posted on Canvas**Acetylsalicylic Acid (Aspirin)**

Assignments (B' – J')	Expected Chemical Shift Range (ppm)	Chemical Shift (Observed ppm)
		169 & 170
		152
		125 – 135 (4 peaks)
		122
		20