Template – copy by hand into notebook

Name _____ Partner Name _____

TA Name _____ Section Letter _____ Day _____ Time _____

Experiment 3 Worksheet – Oxidation of Benzhydrol

Each student submits this individually on Canvas after lab

Pre-Lab Requirements

1. **Dress for lab** – see safety rules – please arrive a few minutes early
2. **Lab Notebook:** copy templates below into designated notebook
   - **Purpose, scheme,** and **reagent table**
   - **Procedure Diagrams** – copy templates provided, follow instructions to complete diagrams

A. Experimental Purpose and Oxidation 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
Benzhydrol	-					1		
Bleach (~0.7 M)		-						
Tetrabutyl ammonium sulfate, $Bu_4N^+HSO_4^-$								
Ethyl Acetate						M		
Benzophenone (product)	-					-		

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

- Bleach & $Bu_4N^+HSO_4^-$ (reagents): divide the mmol of reagent by the mmol of benzhydrol

- Ethyl Acetate (solvent): approximate concentration = divide the mmol of benzhydrol by the volume of solvent

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

C. Procedure Diagrams – *worksheet = template / outline to copy by hand into lab notebook*

- Use the **procedure from the lab PDF** create your hand-drawn experimental instructions
  - Simple sketches & labels for all **equipment, chemical names** with **amounts**, & **transfers**
  - Include clean-up & safety notes throughout your procedure and leave space for observations
- 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 3 website** - Equipment & Safety pages; pictures & videos of the whole lab
- The **class notes** include useful diagrams as well
- *Use as many pages as needed* - at least 3 pages is typical

1. **Reaction Preparation and Set-up** – chemicals added to flask, preparing TLC plates with standards
2. **Monitoring Reaction Progress –** representative aliquot from reaction and steps for spotting, running, and visualizing the TLC plate
3. **Reaction Work-Up** – include all transfers from one container to another
4. **Analysis** – steps for preparing IR sample and rough sketch of both IR spectra; NMR not included in this section

E. Data

Mass of benzhydrol _____ mg

Theoretical yield _____ mg

Calculation:

Sketches of TLC plates and calculated R_f values for each spot:

Standards

Reaction Aliquots (portions over time)

Notes on potential Product loss:

Product mass _____ mg

Percent recovery = (product mass) / (theoretical yield) x 100% = _____%

IR Analysis – Observe the IR spectrum in the website and identify any signals within the expected range. It is acceptable for a signal to be "not observed."

Benzhydrol

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

Benzophenone

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

^{1}H NMR analysis of spectra (Canvas)

Benzhydrol – add structure with H's labeled **A**-**E**

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

Calculations for expected chemical shifts:

Benzophenone structure with H's labeled **A'** – **C'**

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

Calculations for expected chemical shifts:

F. Experimental Methods Writing Worksheet – provided in lab ☺

1. Draw the **reaction scheme** by hand (no copy/paste) and list the **name of the product**.
The reaction scheme includes reactant, reagents over arrow, solvent under arrow, and product.

2. What **glassware** and **equipment** was used for this reaction (aside from chemicals)?

3. How much **benzhydrol** was used? Convert **mass** to **mmol (xx g, xx mmol)**. Show your work, including units with every value. *Calculate or look up the molecular weight of benzhydrol (g/mol) = (mg/mmol).*

4. How much bleach (**NaClO**) was used and what was the **concentration (____ M, ____ mL)**? Fill in the blanks and calculate the quantity of **bleach in mmoles.** Show your work. *Recall Molarity = (moles / Liter) … M = (mol / L) = (mmol / mL).*

5. How much *tert*-butylammonium hydrogen sulfate (**Bu_4NHSO_4**) was used (**xx g**)? This is a catalyst – include only mass not mmol.

6. Determine the **limiting reagent** then calculate the **theoretical yield** (mmol and mg). Show your work, including units with every value. *Determine the mole ratio in the reaction (x mol benzyhydrol / x mol benzophenone). Calculate or look up the molecular weight of benzhydrophenone (g/mol) = (mg/mmol).*

F. Experimental Methods Writing Worksheet (cont'd)

7. What **solvent** was used in the oxidation reaction and in what **volume**?

8. What was the **reaction temperature** and **time**? Was the reaction stirred, refluxed, or standing?

9. What **technique** was used to monitor **reaction progress**? What **solvent(s)** were used during this analysis?

10. List the **identity** and **quantities** of the **chemicals (xx mL)** used in the **reaction work-up**. *Note: quantity of drying agent need not be included.*

11. What **additional processes** were involved in the final isolation of product?

12. What is the yield of **benzophenone (_____ g, _____ mmol, _____ % yield)**? Fill in the blanks and show your work below, including units on every value.

(a) Convert **benzophenone mass** (300 mg) to **mmol** using molecular weight (g/mol) = (mg/mmol).

(b) Calculate **percent (%) yield** using 300 mg as the actual yield and the th. yield from #6.

$$\% \text{ yield} = \frac{\text{actual yield (mg)}}{\text{Theoretical yield (mg)}} \times 100\%$$