Part D: Experimental Methods and Compound Characterization

Experimental methods and compound characterization are found at the end of scientific journal articles, dissertations, and other technical documents to give the reader instructions on how to recreate the experiment and confirm the structure of the newly synthesized compounds. The format and general content differs depending on the field. Students will include this section at the end of several lab reports using the generally accepted guidelines followed by synthetic organic chemists: one General Methods paragraph followed by one additional paragraph per compound synthesized. ***A sample Experimental Methods section is provided online and contains much more information than CHEM 8M students are expected to include.*** *Use passive voice and past tense.*

General Methods

Reagents and by-products do not get full descriptions but are mentioned in the "General Methods" section with the following statement: "All reagents were commercially available, unless otherwise stated." Typically researchers would then describe how reagents and solvents were purified, but *this does not apply to 8M students*. Next, define the abbreviations and list the specifications for NMR (MHz of instrument) and IR (medium for analysis, such as salt plates or Teflon) only if used in the experiment.

Experimental Methods

Following general methods, each organic compound or reaction gets its own paragraph (one paragraph per reaction/compound). Depending on the forms of analysis available to students (based on experimental techniques as well as spectra provided), some or all of the following should be included in the experimental methods and compound characterization section.

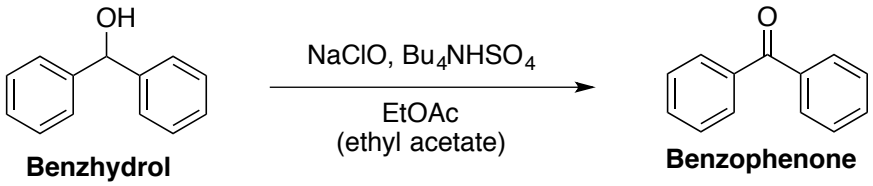
- Reaction scheme - including reactants, reagents, products, solvent(s), and % yield
  (structures and reaction schemes can be hand-written)
- **Full chemical name of product in bold** (common and/or IUPAC)
- Brief description of reaction set up and workup including…
  - Names and amounts of each reactant and reagent (mmol and mL or mg)
  - Name and amount of solvent (mL)
  - Order of addition, if pertinent, and reaction conditions (time, temperature)
  - Description, name, and amount of product obtained and % yield:
    - Ex. "Benzhydrol was obtained as a clear liquid (1.00 g, 87% yield)."

Characterization follows in the same paragraph (after reporting the yield) and includes some or all of the following.

- ^{1}H NMR data – not included in 8M reports, students do not obtain their own NMR's
- Melting point or boiling point
- Optical rotation
- Distinctive IR stretch(es) – one or two distinguishing peaks, such as carbonyl or O-H stretches

Oxidation of Benzhydrol (Exp 3)

Consider the reaction below. Read the procedure then use the worksheet that follows to write the experimental methods section for Exp 3. You may also refer to the sample online.



Benzhydrol **Benzophenone**

EXPERIMENTAL PROCEDURE

Reaction Preparation and Set-up: TLC will be used to monitor reaction progress. Prepare TLC standards and plates before setting up the reaction. Make solutions of the standards (benzhydrol and benzophenone) in small test tubes. This does not require careful measuring, but do be conservative. Dissolve a small amount of the compound (microspatula tip) in ethyl acetate (EtOAc, 1 mL). Obtain three TLC plates, carefully handling by the edges without bending, and gently spot the plate at the origin with a capillary tube (not a melting point capillary). Create one lane for benzhydrol or benzhydrol and leave a space for the reaction mixture to be spotted later (2 spots per plate).

In a 25-mL Erlenmeyer flask equipped with a magnetic stir bar, add 0.37 g (± 0.01 g) of benzhydrol, 5 mL of commercial bleach (approximately 0.7 M NaClO), 5 mL of ethyl acetate (EtOAc), and 40 mg (± 5 mg)* of tetrabutylammonium hydrogen sulfate (Q^+X^- or $Bu_4N^+HSO_4^-$). Secure the flask to a ring stand, loosely stopper, and *stir vigorously on a stir plate without heat*. Increase the stir speed if two layers are observed.

Monitoring Reaction Progress: After about 10 minutes, stop stirring to allow phase separation and remove a small aliquot of the upper layer of the reaction by touching the tip of a capillary tube to the top of the reaction solvent. Spot the TLC plate with this aliquot using a capillary tube alongside the standards. Run the TLC plate using the chambers provided in the fume hood. *Do not remove the chambers from the fume hood!* Develop the plate with a UV or fluorescence light after evaporating the solvent from the plate in the fume hood.

If starting material is still present in the reaction, continue stirring for another 10 minutes and take another TLC aliquot. A faint spot for benzhydrol may still appear on a visualized plate, even when the reaction is complete. When there is no *dark* spot for benzhydrol in the reaction mixture, you may consider the reaction to be complete. The 10 minutes is counted from the first aliquot (20 min total). By the time you run the first TLC plate, it's probably time to run the second! Continue taking aliquots at 10-minute intervals until the reaction is complete. If the reaction is taking longer than 40 minutes, stop the reaction and proceed to *Reaction Work-Up*.

Reaction Work-Up: Transfer the completed reaction mixture to a screw-cap test tube and remove the aqueous layer with a pipet. Wash the organic layer with 3 mL of brine (sat. NaCl) followed by a wash with 2 mL of water – mix, invert, then remove the aqueous layer after each portion of brine or water is added. Dry the organic layer over $MgSO_4$, gravity filter using a pipet with cotton plug, and collect the filtrate in a pre-weighed 25-mL round-bottom flask (RBF). Concentrate using a rota-vap and weigh the product. Pro-tip: the product rarely crystallizes in the rota-vap bath. When the solvent appears to have evaporated, take the flask off the rota-vap and swirl in the ice bath to crystallize. You can still proceed with the product in liquid form.

Analysis: Obtain the IR of the product. Record the identifying peaks in your notebook. Sketch the final TLC plate into your notebook and calculate the R_f values for each spot.

Experimental Methods Worksheet

General Methods: All students will have the following sentence as the entirety of the General Methods section for this report.

"All reagents were commercially available. IR spectra were carried out on NaCl plates with v_{max} in inverse centimeters."

Experimental Methods - specific to each reaction. In future experiments, there will be multiple reactions to report in separate paragraphs.

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

How much **benzhydrol** was used? Convert **mass** to **mmol** and report both **(xx g, xx mmol)**.

How much bleach (**NaClO**) was used and what was the **concentration (xx M, xx mL)**? Since the chemical is a solution in water, the molarity must be included.

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

What **solvent** was used and in what **volume**?

Was the reaction heated, cooled, or room **temperature** (25 °C)? What was the reaction **time**? Was the reaction stirred, refluxed, or standing?

How was the **reaction monitored** for completion? What **solvent(s)** were used during this analysis?

List the identity and quantities of the chemical(s) (xx mL) were used in the **reaction work-up**.

How much product was isolated (xx g, xx mmol, xx % yield)? Convert product **mass** to **mmol**. Calculate the theoretical yield to determine % yield. Only **% yield** should be reported.

- Sample data: **0.30 g** of product

Now put it all together in complete, concise sentences to write the experimental methods section. Show this to your TA, who will provide feedback and likely send you back for a re-write.

Part E. Format for Literature References

There is a standard A.C.S. (American Chemical Society) format for listing references in the chemical literature that you are required to follow (http://pubs.acs.org/books/references.shtml). This format, illustrated below, must be used in the reference section of your report, if appropriate. Be sure to document all assertions and past work described in your reports with a footnote. Footnotes can be referred to more than once. Use superscripts with corresponding numbered references at the bottom of the page or at the end of the report.

BOOKS

Author's last name, first initial, *Title of Book,* Publisher: City of publication, **Year of pub**.; pages used.

Examples

Crews, P.; Rodríguez, J.; Jaspars, M. *Organic Structure Analysis, 2nd Ed.;* Oxford: New York, **2010**; pp. 67-70.

Palleros, D.R., *Experimental Organic Chemistry;* Wiley: New York, **2000**; pp. 61-70.

JOURNALS

Author's last name, initials.; 2nd author's last name, initials.; (continue for each author). *Journal abbrev.* **Year**, *Vol.,* first to last page of article.

Proper journal abbreviation used in italics*, **year in bold, *volume in italics*, no issue number

Examples

Tansakul, C.; Lilie, E.; Walter, E. D.; Rivera III, F.; Wolcott, A.; Zhang, J. Z.; Millhauser, G. L.; R. Braslau, R. *J. Phys. Chem. C,* **2010**, *114,* 7793-7805.

Sanchez, L. M.; Lopez, D.; Vesely, B. A.; Della Togna, G.; Gerwick, W. H.; Kyle, D. E.; Linington, R. G. *J. Med. Chem.*, **2010**, *53*, 4187-97.

Woehrmann, M. H., Gassner, N. C., Bray, W. M.; Stuart, J. M.; Lokey, S. *J. Biomol. Screen.* **2010**, *15,* 196-205.

WEB SITES

Use full website addresses to allow the reader to locate referenced material on the web. **Be wary of the content. The info on the web is usually not peer reviewed, and can be erroneous!** If you do cite a website, include the date the website was accessed.

Example

http://organicchemistry.wordpress.com/2007/08/18/tips-for-writing-organic-chemistry-lab-reports/ accessed 7-23-09.