

# CHEM 321: Guidelines for writing the Experimental section of a lab report

**GOAL:** A scientist familiar with common laboratory operations could use your "Experimental" section to repeat successfully what you did in the laboratory.

The following seven format requirements must be met. A report that has serious format flaws will be returned to you as "dead on arrival". The source of the protocol you used must be properly cited.

## 1. Focus on the scientific goals, not on procedure or sequence

The temptation is to recount the procedures and sequence of events that you followed, but this distracts from the readability and scientific usefulness of a report. Before you begin writing, make sure that you can easily and accurately describe both the generic goals and the scientific goals of the experiment. Then, ask how did each step serve the goals of the experiment? Describe operations or treatments in light of the goals so the reader can understand the scientific logic behind them. As you are writing the experimental section, it is important to keep the product of the reaction (or isolation) in mind. The focus of each sentence should be the reagents used to synthesize the product (or in the case of an isolation, the starting materials), or the location of the product, or the product itself.

For example, next semester, you will be synthesizing an alcohol using a Grignard reaction.

**Wrong:**  $\text{NaHCO}_3$  (5 %) was added to the organic layer, and anhydrous  $\text{CaCl}_2$  was added to the crude product.

**Right:** The organic layer was washed with sufficient  $\text{NaHCO}_3$  (5 %) to neutralize acid and dried over anhydrous  $\text{CaCl}_2$  (1 week).

Notice, in the right sentence, "the organic layer" is the focus of the sentence (because this is where the main product, the alcohol, is at that moment in the experiment), and this sentence focuses on the science behind the raw procedure. It tells the reader exactly what is the purpose of the wash. As you may remember from general chemistry, sodium bicarbonate ( $\text{NaHCO}_3$ ) was used to clean up/neutralize acid spills.

## 2. Write a history (past tense) of exactly what YOU did using passive voice.

**Wrong:** I mixed 20.09 g of cyclohexanol with 6 mL of 85 %  $\text{H}_3\text{PO}_4$ . (Don't use first person active)

**Wrong:** Mix 20.09 g of cyclohexanol with 6 mL of 85 %  $\text{H}_3\text{PO}_4$ . (Recipe format – not historical.)

**Right:** Cyclohexanol (20.1 g, 0.201 mol) was mixed with  $\text{H}_3\text{PO}_4$  (85 %, 6 mL). (Third person passive, past tense)

Be concise but not "choppy". Do NOT use the word "then" unless absolutely necessary (usually, the sequence of events will be obvious). Write an accurate history. What you *actually did* may not be what the protocol said to do, and your results may not be what the protocol said to expect. If there were significant deviations from the protocol, tell what YOU actually did. (Eventually, in future reports, you will describe procedural mistakes in the Discussion, where you will also tell what effects they might have caused.)

## 3. DO NOT WRITE A RECIPE (see "wrong" example above) - appropriate only for Lab Notebook.

## 4. Report amounts of reagents using the following style. Names of compounds are the subject(s) of the sentence. The mass and/or volume and number of moles of reactants are reported in parentheses. Separate numbers from unit symbols with a space. Do not report moles of solvents.

**An example with reagents and a solvent:**

Cyclohexyl bromide (10.0 g, 0.060 mol) and KOH (20.0 g, 0.357 mol) were mixed with ethanol (95%, 50 mL).

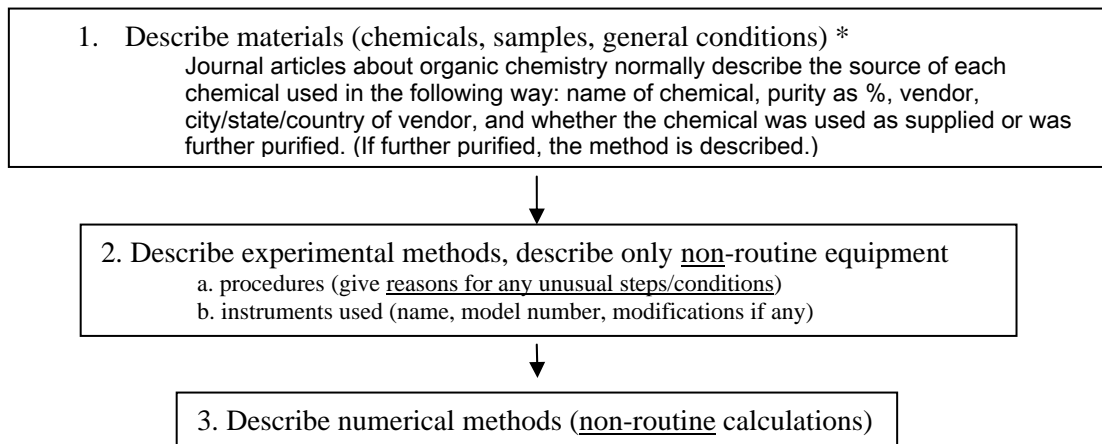
**An example with a molar concentration:**

NaOH (3 M, 8 mL) was added with good mixing to dissolve all the small crystals.

5. Review the “Common Techniques Used in Organic Chemistry” flowchart and your lab notebook to recall names of “common” operations used in the experiment you just completed. (*Your notebook but not the report should contain written definitions for all that are new to you.*) Use these names in your report, but do not describe the operations or routine glassware needed (assume that your reader knows this information).
6. Do NOT include any results. Do NOT include a table of physical properties (that's for your notebook). Do NOT show computations in this section, but *do show them in an Appendix* (See inside “Guidelines for Results, Discussion, and Appendix Sections of Lab Reports” document on course website).
7. **Miscellaneous (as needed):**
  - **Special reagents, special glassware, or non-routine operations:** identify and tell why they were used (*see #4 above for what to omit!*)
  - Length of reaction time; reaction temperature
  - Any unexpected observations
  - Protocols (including reactions) used for chemical characterization of products

### EXAMPLES FROM THE PROFESSIONAL LITERATURE

Professional reports of experimental work are organized as follows [adapted from M.S. Robinson, F.L. Stoller, J.K. Jones, and M.S. Costanza-Robinson, Write Like a Chemist (pilot draft 2005-2006)]:



*\*For the purpose of this course, you may omit box 1 when you write your reports.*

Two short Experimental sections from articles in a professional journal follow (in a different font). Try to emulate their style as you organize and write your Experimental section. Note in particular how they are organized, how concise they are, what they assume the reader knows, and the rare use of the word “then”. [*Even in these examples, there are a couple of grammatical and format errors – can you find them?*]

*From M. E. Squillacote and F. Liang, J. Org. Chem., 70 (17), 6564 -6573, 2005. (An introductory sentence, not shown here, indicated the sources of chemicals. The selection is taken from the part of the report that describes how needed compounds were synthesized.)*

3,4-Dimethyl-3,4-hexanediol: A solution of mercuric chloride ( $\text{HgCl}_2$ ) (10.5 g, 0.0387 mol) in 2-butanone (63.0 g, 0.875 mol) was added slowly to magnesium turnings (10 g, 0.42 mol) in 120 mL of dried benzene until the reaction mixture began to reflux spontaneously. The remainder of the solution of mercuric chloride in 2-butanone was added over a 45-min period at such a rate that a vigorous reflux was maintained. After the reaction mixture had been refluxed for an additional 1.5 h, 40 mL of distilled water was added and the mixture was heated to reflux for 30 min.

The gray solid which formed upon addition of water was separated from benzene solution by filtration and then returned to the reaction flask, together with 100 mL of fresh benzene. This mixture was refluxed and filtered. After the benzene filtrates were combined and concentrated, 16.11 g of oil (25.2% yield on starting ketone) was obtained. NMR spectra showed it was an approximately 1:1 mixture of meso and DL isomer of the pinacol 3,4-dimethyl-3,4-hexanediol (bp 108-114 °C, 32 mm).

From A. Srivastava, S. Ghorai, A. Bhattacharjya, and S. Bhattacharya, *J. Org. Chem.*, 70 (17), 6574 -6582, 2005. Chemical names were previously equated to **bolded** numerals, which are used here to stand for the corresponding name.

## Experimental Section

**General.** All reagents were purchased from best-known commercial sources and used without further purification. Silica gel 60-120 mesh size was used for chromatography. FT-IR was performed as KBr pellets of solid **1**, and xerogel of **1**, as well as a neat solution of **1** in MeOH. Melting points are recorded in open capillaries and are uncorrected. UV-vis experiments were performed with the spectrophotometer whose cell holders were connected to a water bath for maintenance of desired temperature. UV irradiation of the gel was done by a UV lamp with emission around 350 nm. pH measurements were done via a precalibrated pH meter.

**Azobenzene derivative, 3.** A mixture of bis-1,2:5,6-diisopropylidene-glucosyl derivative **2** (5.00 g, 7.50 mmol), Zn (0.99 g, 15.11 mmol), NaOH (0.61 g, 15.25 mmol), EtOH (60 mL) and H<sub>2</sub>O (15 mL) was heated under reflux for 3 h, and then cooled to 25° C and filtered. The filtrate was concentrated under reduced pressure and the residue was extracted with CH<sub>2</sub>Cl<sub>2</sub>. The organic layer was washed with water, dried and concentrated. The residue was chromatographed over silica gel (100-200 mesh size; EtOAc-petroleum ether, 1:4) giving **3** (2.38 g, 50%) as an orange foam,  $[\alpha]_D^{25} -5.4^\circ$  (c 0.45, CHCl<sub>3</sub>); IR (KBr, cm<sup>-1</sup>): 1610; . . . [characterization data continue]