ACS-Style Final Reports

(Revised Spring 2007)

As with any other field, the practice of chemistry involves the daily use of oral and written communication skills. Like it or not, your professional success will depend very heavily on your ability to write and present papers, reports, proposals, evaluations, etc. Thinking up or carrying out a clever experiment won't bring great rewards if you cannot clearly and convincingly communicate your ideas and discuss your results.

Scientific publications are written in a more formalized "journal" style. You are now writing for a well-educated audience, which includes B.S., M.S., and Ph. D. chemists. Therefore, you can assume that your audience will know basic reaction set-ups. You need to raise the level of presentation considerably, and at least give the appearance that you know what you are doing. With the proper writing style, one can even take a completely failed experiment and make it appear that the results have left the world a little more enlightened through a sound scientific analysis of the results.

Before you start, write a "back-of-the-envelope" outline of how you plan to organize the manuscript, including the critical first "intro" sentence to each paragraph. For inspiration, be sure to read the original experimental background (including all references in the handout), experimental, and discussion of the experiment as provided to you originally.

These reports must be produced by a word processor, be double-spaced, and include structures and reaction schemes drawn using ChemDraw. To download ChemDraw, go to http://scistore.cambridgesoft.com/sitelicense.cfm?sid=86 and type in your PSU email address to download the software. See below for complete word processor specifications.

For Chem 36H final reports, please include the following components or sections in the order given:

1. Title
2. Byline and Affiliation: Author name(s), e-mail addresses and Place where research took place (Department of Chemistry, The Pennsylvania State University)
3. Abstract
4. Introduction
5. Results & Discussion (combined)
6. Conclusions (include ideas for future work)
7. Experimental Section – start this section with a paragraph describing general items like how solvents were purified, where chemicals were purchased from, the type of instrumentation used, etc. This paragraph is followed by an experimental procedure for each compound made (similar to the previous assignments); see a *J. Org. Chem.* article as a guide.
8. Acknowledgements
9. References

10. Supporting Information – copies of annotated spectral data and photocopies of all referenced materials, including the experimental handout, graded Experimental Outline, and in-lab notebook pages.

The final report must be computer-generated, double-spaced, 12-point font, Times New Roman font, 1" margins, full justification, and inserted page numbers except for title page. Remember to attach the grade sheet to the front of the report. The final report must be submitted in a presentable manner; place it (stapled) in a two-pocket folder.

You are required to use a chemical structure drawing program; ChemDraw is preferred. For correct drawing parameters in ChemDraw, click on "Object" from the menu bar, select "Apply Object Settings From" and choose "ACS Document 1996". When typing text in ChemDraw, use 12 point font, Times New Roman. Be sure to select "Fixed Lengths" and "Fixed Angles" under "Object" when constructing structures. If you need to use Greek letters/symbols, within a text box, type in the corresponding English letter, highlight the English letter, then select "Symbol" under the font type; the "Symbol" font provides Greek letters/symbols. Number each organic compound/intermediate in a scheme. Note that the numbers are in bold font. Be sure to keep the numbers of compounds the same throughout the report.

Detailed Information on Sections of Report

Abstract

"The abstract allows the reader to determine the nature and scope of the paper. The optimal length is one paragraph long, but it can be as short as two sentences. The length depends on the subject matter and the length of the report – between 80 and 200 words is usually adequate. Briefly state the purpose of the project, indicate the theoretical or experimental plan used, summarize the principal findings, and point out major conclusions. Do not cite references, tables, figures, or sections of the report. Do not include equations, schemes, or structures. Write the abstract last to be sure that it accurately reflects the content of the paper." (The ACS Style Guide: A Manual for Authors and Editors, 2nd Ed" by Janet S. Dodd, published in 1997)

Introduction

"A good introduction is a clear statement of the problem or project and the reasons that you are studying it. This information should be contained in the first few sentences. Give a concise and appropriate background discussion of the problem and the significance, scope, and limits of your work. Outline what has been done before by citing truly pertinent literature. The introduction can be one or two paragraphs long. " (The ACS Style Guide)

The Introduction demonstrates to the reader that you really know what you are talking about because of your background knowledge and reading. It does NOT contain a step-by-step description of every experimental detail, but rather talks about the chemistry in broader terms. In general, for each intermediate or product, you should cover:

- The class of compounds you are synthesizing and isolating (for example, an alkene, organometallic compound, ester, heterocycle, etc.) and why compounds in this class are important from a commercial or biological standpoint. Give some important uses of the target substance or of compounds in its class or the reaction that led to its production.
- The type of reactions you are using to synthesize it (for example, elimination, oxidation, reduction, condensation, acid chloride, coordination complex formation, etc.) or methods you are using to isolate it from a natural source (for example, extraction, chromatography, etc.). Give any interesting historical information on the discovery or the development of the reaction(s) or method of isolation.
- Are these methods suitable for all cases? For example, can you use chromic acid to oxidize all alcohols, 1°, 2°, and 3°, to ketones? How would results vary as the structure of the starting material changed? Would both liquid and solid starting materials be handled the same way?
- What variations are available in terms of chemical reagents such as oxidizing agents or catalysts that would yield similar or improved results? For example, can both sulfuric acid and phosphoric acid be used for acid-catalyzed dehydration of an alcohol and does phosphoric acid offer some advantages over sulfuric acid?
- Are there any special methods or techniques that are noteworthy? For example, the use of an acidic resin as an acid catalyst makes the isolation of the product in an ester synthesis much easier. Or the isolation of saturated fatty acids can be accomplished by using shape-excluding clathrates.
- Molecular Modeling – What questions might be answered by Molecular Modeling?

Examples:

Tetraaryl Porphyrins and their metal complexes - Discuss UV/Vis spectra

S vs. O metal ligand bonding in DMSO – Discuss metal complex structures by IR.

Benzpinacol photochemical formation -Discuss promoting electrons and

radical coupling (singlet triplet states)

Ferrocene - Discuss superaromaticity of ferrocene and its aromatic like

behavior on electrophilic substitution.

Background information is available from your handout, handout references, your lecture course text, or additional books and articles in the chemical literature.

Many good examples of introduction sections are to be found at the beginning of the many synthetic experiments in a lab text such as K.L. Williamson's Macroscale and Microscale Organic Experiments.

Introduction Comments:

a. Don't capitalize chemical names.
b. Use Schemes for reactions, use Figures for a single compound.
c. Include structures, reactions and 3-D structures. Readers want to see structures reactions and mechanisms. Do not tape in photocopied reaction schemes – this will not be accepted and points

will be deducted.
d. Your general and organic chemistry lecture texts, for example Brown & Lemay, Jones, or McMurry, are acceptable references.

Results & Discussion

Results: "Summarize the data collected. Include only relevant data, but give sufficient detail to justify your conclusions. Use equations, figures, and tables only where necessary for clarity and brevity." (The ACS Style Guide)

Example: Synthesis of $TiCl_4$

Titanium tetrachloride was prepared by passing chlorine over a mixture of titanium dioxide and carbon maintained at a temperature of 550°C. The pale yellow liquid product was collected in an overall yield of 75% and characterized by its boiling point, color and conversion to a complex with acetyl acetone.

Results sections should be short. Instead of saying: "The a-pinene was reacted for 1 hr with BH3-THF complex at 60°C and then sodium hydroxide and hydrogen peroxide to give 0.23 g of isopinocampheol as a white crystals...." Write in concise generic terms: "Isopinocampheol was produced in 37% yield by oxidative hydroboration of a-pinene and characterized by GC-MS."

Discussion: "The purpose of the discussion section is to interpret and compare the results. Be objective; point out the features and limitations of the work. Relate your results to current knowledge in the field and to your original purpose in undertaking the project. Suggest further study or applications if warranted." (The ACS Style Guide)

The Discussion should detail the success of your synthesis or mol. modeling calculations, giving reaction mechanisms where relevant. If your yield was low, you should explain why by using sound scientific reasons (not "I dropped the flask") and give suggestions as to what could be done to improve the synthesis if you repeated it, etc. The discussion should also contain a detailed interpretation of your spectral or chromatographic data. Here is where you should discuss the spectra in as much detail as required to prove unambiguously that your compound has the desired structure and purity giving specific assignments to NMR peaks, explaining mass peaks, etc. and the relation of spectra to structure or structures to GC peaks. Mass spectral fragmentations must be reasonable. You may wish to organize this into subsections of Synthesis (or Isolation), Characterization, and Molecular Modeling. The discussion section should finally close with a short one-paragraph summary of what you have done and your important results and conclusions.

You will lose points if you go on about "This reaction was successful because I got something etc." It is hoped and assumed that it is successful simply because it is being submitted to be published.

If you have improved yields by modifying conditions, this should be discussed positively. For example:

> "Initially, the yields of the reaction were lower than reported by Holz and Elder. Therefore, the heating rate and time were varied, and this modification was monitored by TLC. By extending the heating time to 2 hrs, improved yields were obtained."

Do NOT write:
"The amount of crystals gotten was disappointing and so I tried something different".

The Results and Discussion Section should not contain experimental details that are described in the Experimental section.

Experimental Section

General Experimental Methods

The Experimental Section normally starts with a brief description of how you obtained your spectra (the type and manufacturer of each instrument, the sample preparation method, calibration methods) and how you carried out your chromatographic separations [the instruments used and type and supplier of GC column (for gas chromatography) or LC column (for liquid chromatography) and TLC plates]. Also, the sources of special starting material chemicals and their purity should be listed here. You don't have to list the source of the common reagents like solvents and drying agents. You can assume that the reader could easily find a source for common chemicals such as dichloromethane, sulfuric acid, ethanol, sodium hydroxide, acetic acid, etc. If any special steps were taken to test the purity of or to purify or dry starting materials or solvents, this should be stated here. Information in this section should be patterned after the Experiment Section in any quality journal article.

Example:

"Spectral Measurements: Infrared spectra were recorded on a Mattson Instruments (Madison, WI) model 1020 FT-IR. Ultraviolet spectra were recorded in ethanol on a Hewlett Packard 5842 Diode Array UV/Vis spectrophotometer. HPLC was carried out on a 10cm x 1.8 mm ID column packed with 10 mm silica (Keystone Scientific, Bellefonte, PA) using a Milton Roy metering pump and a GOW-MAC UV detector at 254 nm. The mobile phase was 60:40 chloroform/hexane flowing at 2 mL/min. Melting points were taken on a Thomas-Hoover melting point apparatus and are uncorrected.

Chemicals: Pyridine was obtained from J.T. Baker Chemical Co. (Phillipsburg NJ). Pyrrole, 4-anisaldehyde, and benzaldehyde were purchased from Aldrich (Milwaukee WI). All chemicals were used as supplied except for the aldehydes, which were distilled under nitrogen just prior to use. Acetaminophen, chloramphenicol, zomepirac, and probenecid (Sigma Chemical Co., St. Louis, MO), Coomassie Blue reagent (Pierce

Biochemicals, Rockford, IL) were used as received, HPLC grade ammonium phosphate (monobasic), acetonitrile, and methanol were from Mallinckrodt (St. Louis, MO).

Note The stockroom is not a manufacturer! You can't write "9-anthraldehyde and TLC plates were obtained from the stockroom."

GC and/or GC-MS should have column length and ID and coating and temperature program. Solvent is unimportant as is carrier gas. Example:
Gas chromatography was carried out on an HP 5980 GC using a 30m x 0.25 mm i.d. capillary column with a 25 micron coating of 5%phenyl/95% methyl silicone which was programmed from 40 to 250°C at 10°/min.

Synthetic Procedure for each Compound Made

The second part of the Experimental Section contains an extremely concise and well-organized description of the synthetic procedures. If the synthesis(es) you are describing involves more than one step, you should list the preparation of each intermediate in a separate entry.

Journal production is costly today and authors often pay hundreds of dollars per page to publish their research. In order to hold down costs and therefore make journals more affordable, every effort is made to be concise in preparing manuscripts. To quote from the Guidelines for Authors for the Journal of Organic Chemistry:

> "Complete descriptions of apparatus and procedural details should be avoided unless precise adherence to a protocol or type of apparatus is essential to the success of a reaction being described."

This means that you don't have to put in every specific piece of glassware such as 100-mL beaker or 50-mL Erlenmeyer flask, but can assume the reader can figure out what to use.

The following example from the Guidelines shows the difference from what an inexperienced student might write and what the journals would prefer:

_ TOO MUCH DETAIL:

> "Into a flame dried, 100-mL, three necked, round-bottomed flask equipped with a magnetic stirring bar and a nitrogen gas inlet was placed a solution of 9.6 g (0.1 mol) of cyclohexanone in 50 mL of ether. To the stirred solution, under nitrogen, was added 3.8 g (0.1 mol) of lithium aluminum hydride. The solution was refluxed for thirty minutes."

_ PREFERRED STYLE: "A solution of 9.6 g (0.1 mol) of cyclohexanone and 3.8 g (0.1 mol) of $LiAlH_4$ in 50 mL of ether was refluxed under N_2 for 30 min." _ Note that FORMULAS, i.e. $LiAlH_4$ and N_2 are used instead of the compound names.

_ CORRECT COMMON UNIT ABBREVIATIONS:

g - gram - no period!

mL - milliliter - no period!

mol - mole(s) - no period!

min - minute(s) - no period!

s - seconds(s) - no period!

_ Note that common abbreviations do NOT have a period after them. The ACS Style Guide lists other common abbreviations; it's located on the side bookshelf.

At the end of each synthetic procedure entry, a very compact list of physical properties, including melting or boiling points, and spectral data should be given. An example experimental section from an article in the Journal of Organic Chemistry is given below. Your experimental section must adhere to this format! GIVE YIELDS--grams or mg, and % of theoretical. Gas chromatographic data:

GC: starting material 2, 4.67 min; cis 3, 5.32 min; trans 3, 6.04 min Absolute retention times in GC are not so important since they will vary from GC to GC. Defining the order of elution is more important.

J. Org. Chem. Entry Example: Note when characters are bolded, italicized.

Indoleamide, **2:** The isatin-3-hydrazone (**1**, 3.0 g, 15 mmol) was dissolved and heated in 16 mL of 21% sodium ethoxide in ethanol at 60-70°C for 10 min and refluxed until the evolution of N_2 stopped. After pouring the reaction mixture on ice and acidifying to pH 1 with 10% HCl, the reaction mixture is extracted with ether (2x25 mL), the combined ether extracts dried over anhyd Na_2SO_4, and the ether removed under vacuum to yield a yellow solid. This was recrystallized from H_2O to give 2.1 g (74%) of **2**, mp 123-125°C (lit[3] mp 124-126°C). ^1H NMR (80 MHz, $CDCl_3$): d 2.5 (2H, d, *J*=7 Hz, Ar-CH_2-), 7.6 (4H, m, aromatic); IR (KBr 1730 (C=O), 2880 (N-H) cm^{-1}; MS *m/z* (% rel. int.) 133 (100, M+), 105 (23, M-CO), 91 (32, M-CH_2CO); UV (EtOH) λmax 262 nm (e = 19,200)

Other comments on the Experimental Section:

Don't use the implied "I did something" style, i.e. don't write: "(I) Mixed aldehyde and acid, heated to boiling....., (I) cooled and extracted....." This is OK for your notebook, but not the final report.

Each synthetic step is started in a separate entry headed, in bold, with the compound name (and number, if numbered anywhere in the report) at the beginning.

All quantities must be given: g & mmol for reactants, concentrations of solutions in % or M.

Don't describe glassware unless it is special. The chemist doing your experiment has enough training to be able to figure out what type and size flask to use.

Run-on sentences should be used.

Include weight and % yield, mp and lit. mp for all solids including isolated intermediates.

Sentences cannot start with a chemical formula, for example, Na2SO4.

You shouldn't start a sentence with numerical digits or a formula. Example:

"1 g of NaOH is added" should be "Sodium hydroxide (1.0 g, 0.025 mol) is added to.....

Always put a leading 0 before a decimal place, i.e. 0.25 g, not .25 g.

Acknowledgments

This section gives the author(s) an opportunity to recognize those people who were a help. Examples include Dr. Masters, your TA, Dr. Bortiatynski, and the stockroom ladies, Michele Brown and Linda Price.

References

References appear at the end of the report and should be done as endnotes and must conform to the guidelines as presented in Handbook for Authors published by the American Chemical Society. You can download a Quick Guide to References – ACS Style by accessing http://courses.chem.psu.edu/chem431/Manuscript.html. Only list those references actually used in lab or cited in your Final Report. All published spectra, which you use for comparison, should be referenced.

Supporting Information

ALL Spectra SHOULD BE ATTACHED TO THE REPORT AND REFERRED TO BY FIGURE NUMBER. All UV/Vis spectra must have lmax for all maxima. Point out M^+ and major fragmentations on mass spectra. Draw the full structure on each spectrum and correlate the prominent spectral features with the main functional groups, alkyl groups, protons, etc.

That is, be sure to annotate each peak in ^{1}H NMR data and every key functional group peak in the IR data.

Attach all in-lab notebook pages that were used for the experiment. Be sure all pages are signed by a TA.