

Chemistry 321L: Experimental Writeup Lab Report

Title

The title should be descriptive of the entire project, not just simply “Triphenylmethanol” or “S_N1 Reaction”. If you choose a cute/clever title, make sure it is also descriptive. The title should be on the first page of your experimental section (centered). In the top right hand corner (above the title) make sure you have your full name, Chem 321L, section number, and TA’s name.

Experimental Section

The Experimental Section should be a concise step-by-step summary of what was done during the experiment (the actual procedure) followed by a summary of the physical properties you recorded (melting point, etc) and spectral data (IR, NMR, etc). In general the experimental section should give all the information necessary for someone to actually repeat your experiments successfully.

The Experimental Section is always written in the third person, past tense. Under the Experimental Section, each experiment is described in a separate paragraph that has a brief, descriptive title in bold. (For organic reactions this title is often the name of the compound synthesized.) When mentioning a compound that was used in an experiment, you should write it as follows: compound name (amount in grams, number of moles). It is also important to note concentrations, time a procedure took and any observations that were made (color change, odor, etc).

You are strongly encouraged to look at examples of Experimental Sections from articles in the *Journal of the American Chemical Society* or the *Journal of Organic Chemistry*. When writing the Experimental Section, you may assume that your audience has an organic chemistry background. Therefore, you may refer to procedures like recrystallization, filtration, distillation, etc. without having to describe them in lengthy detail.

Examples of experimental write ups for CHEM 321L:

2,5-Di-*tert*-butylacenaphthene. A soln of acenaphthene (15.1 g, 97.7 mmol) and aluminum chloride (2.65 g, 19.9 mmol) in carbon disulfide (100 mL) were added to *tert*-butyl-chloride (20. mL, 180 mmol) dropwise via syringe over 25 min. The soln was stirred for an additional 3 h, then refluxed for 1 h. Upon cooling, water (100 mL) was cautiously added, followed by concentrated HCl (5 mL) and stirring was continued until all of the dark solid had dissolved. The mixture was extracted with carbon disulfide, dried over Na₂SO₄, and filtered. The solvent was removed under vacuum and the resulting yellowish wax was recrystallized once in glacial acetic acid and then once in toluene/ethanol to yield a white precipitate (7.42 g, 27.8 mmol, 30.3%): mp 161-162 °C.

1,3,5-Tris(cyanomethyl)-2,4,6-triethylbenzene. 1,3,5-Tris(chloromethyl)-2,4,6-triethylbenzene (0.70 g, 2.3 mmol) was dissolved in acetone (60 mL) and heated to reflux. Sodium cyanide (0.50 g, 10.2 mmol) was added. Then, water (25 mL) was added slowly until the soln became turbid. Acetone (2 mL) was added, and the reaction was left to reflux overnight. After 15 h, the soln was poured onto ice water (100 mL), and the product precipitated out as a white powder which was filtered and recrystallized from absolute ethanol yielding 1,3,5-tris(cyanomethyl)-2,4,6-triethylbenzene (0.60 g, 2.0 mmol, 88%): mp 220-222 °C; IR (KBr) 2248 cm⁻¹.

1,4-Dimethyl-2,3,5,6-tetraethylbenzene. *para*-Xylene (2.0 g, 19 mmol) was dissolved in ethyl bromide (20. mL, 29 g, 270 mmol) in a 2-neck 50-mL round bottom flask fitted with a condenser and drying tube. Anhydrous aluminum chloride (1.3 g, 9.9 mmol) was added slowly while the soln bubbled and turned from clear to yellow to orange. After 2 h at reflux, the soln was poured onto ice/water, extracted with methylene chloride, dried with anhydrous sodium sulfate, and evaporated yielding a white solid, 1,4-dimethyl-2,3,5,6-tetraethylbenzene, which was recrystallized from absolute ethanol (0.72 g, 3.3 mmol, 18%): mp 114-117 °C.

Worth noting from above (Your grade will be better IF you follow these!):

- ✓ Really check the format in the examples above and use it!
- ✓ There is a period after each title (from each reaction) and the title is bold.
- ✓ The first word of the title is capitalized unless it is italicized (*para*, *tert*, etc.).
- ✓ Compound's names (including reactants, products, solvents, etc) are not capitalized unless they are at the beginning of the sentence.
- ✓ There are spaces between amounts and units (2.0 g not 2.0g), except when using percent recovery (18% not 18 %).
- ✓ Significant figures are important for reactants and any solvents that could play a limiting role.
 - For the example above for **2,5-Di-*tert*-butylacenaphthene**, acenaphthene, aluminum chloride and *tert*-butyl-chloride could all potentially be limiting so their significant figures are important. However, carbon disulfide, water, and HCl cannot play a limiting role (they are just acting as solvents, etc) so their values aren't reported in terms of significant figures.
 - When reporting the percent yield value (i.e., (7.42 g, 27.8 mmol, 30.3%)), these values are significant and must be with the same number of significant figures as the limiting reactant. Therefore, you must know what the limiting reactant is!
 - Please review how to determine the number of significant figures from your general chemistry book!
- ✓ The melting point should be a range and ONLY given IF you actually recorded one.
- ✓ Dropwise is one word.
- ✓ The color of the final product is important as are any color changes observed during the reaction.
- ✓ If you use a number that starts with a decimal, put a zero in front of the decimal (.70 becomes 0.70).
- ✓ mL is not the same thing as ml; the L should be capitalized.
- ✓ Some common abbreviations that **MUST** be used are:
 - h for hour(s); min for minute(s); soln for solution