Written Tutorial Assignment

T. R. Hoye

Chemistry 4311: Advanced Organic Chemistry Laboratory

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To meet the Writing Intensive (WI) requirement of Chem 4311, I am asking you to prepare a short document (600-800 words; FYI, there are 980 words on this sheet)—let’s call it a written tutorial. The topic will be related to one of the various techniques with which you have gained experience through your work in the lab this semester. As you write, imagine your audience to be the group of future students who will take Chem 4311. Prepare a written tutorial that those students will find useful to enable them to even more quickly assimilate your topic technique into their routine work. In other words, what else would you like to have been told that would have made your mastering of that technique go more smoothly? Strive to make your document clear, concise, and useful. I have identified five potential topics. As you can see, I have asked some leading questions and/or given some specific suggestions of things to be included. Please address as many as you can in your written tutorial. You may include other issues if you deem them important, but there isn’t a need to do so. However, try to avoid duplicating the “nuts and bolts” information that is provided in the Zubrick text and in the TA-run tutorials related to the technique. I have (randomly) assigned you the topic outlined below. Because I have delayed so long in getting this assignment to you, rather than assigning you a specific topic, I will let YOU CHOOSE which ONE topic to write about.

Evaluation: Your written tutorial will be evaluated on the basis of its effectiveness as a potential document that we might actually use in a future offering of the course. Thus, content and clarity of the writing are both important. I anticipate using only two categories of evaluation: very good (90 out of 100 points) and excellent (100 out of 100 points). In other words, every paper will receive one or the other of these two scores.

Topic: Thin Layer Chromatography

What observations indicate when a tlc plate is overloaded with sample? What step(s) can be taken to a) verify and b) minimize and solve overloading situations?

How is an ideal solvent system identified by which to a) monitor reaction progress and b) select the solvent to be used in an MPLC separation of two compounds of similar polarity? Describe the chemistry that occurs on the tlc plate by which "UV active" compounds can be located. What caveats should be considered when interpreting the intensity of such "spots?"
**Topic: Infrared Spectra**

Write a set of instructions for using the Midac FT-IR spectrometer. Imagine that what you produce will be laminated and posted beside the spectrometer. Discuss a) sample preparation, b) the necessity (or lack thereof) of collection of a background/reference spectrum, c) how to optimize the sample size so that the maximum peak intensity does not "overload" the spectrometer (i.e., so that no peak has an intensity of <10% transmission), d) how to name a file so that it can be identified later, and e) how to print the entire spectrum and an expanded portion of the spectrum--say that between 1400 and 1800 cm⁻¹.

**Topic: Instructions for Processing 1H NMR Spectral Data from the SGI Workstation**

Write improved instructions for using the SGI workstation to access, process, and print NMR spectra. Imagine that what you produce will be laminated and posted beside the SGI. Indicate how to find the proper file from the information on the printed spectrum that is routinely provided and from the information in the blue NMR binder. Detail how to load, phase, reference, integrate, and print a spectrum. Describe how to expand, determine an appropriate vertical scale, and print a portion of the spectrum, including the peak positions, corresponding to a single multiplet. Explain how to determine the distance in hertz (Hz) between any two lines in the spectrum.

Note: Your paper should NOT simply be a list of commands. Rather it should include enough detail to give a sense of what each command does.

**Topic: Capillary Gas Chromatography**

Describe the essential features (dimensions, film thickness, and liquid phase structure) of the capillary gas chromatography column that we are using. FYI, the fused-quartz silica capillary column is coated with a layer (film) of HP-5, a silicone based polymer. This column tends to separate components on the basis of boiling point. Describe why/how. Discuss why the separation strategy nearly always uses a temperature program that ramps the oven temperature from, e.g., 50°C up to some method-defined maximum temperature. Describe why the retention time of two components, A and B, is 6.07 and 10.43 minutes vs. 6.07 and 10.14 minutes when the same sample is run on the same column with the following two methods, respectively: i) starting T = 50 °C; initial hold time = 2 min; ramp rate = 20 °C/min; final T = 200 °C i) starting T = 50 °C; initial hold time = 2 min; ramp rate = 20 °C/min; final T = 250 °C
**Topic: Medium Pressure Liquid Chromatography (MPLC)**

Discuss the relationship between tlc retention factor (Rf) and the number of column volumes it takes to bring the same compound off of an MPLC column eluted with the same solvent. Discuss how to estimate the column volume of an MPLC column packed with silica gel. Advise how to decide what fraction size (volume) to take vis-à-vis the column volume. Describe why it is wise to use a column that has previously been flushed with a more polar solvent than the one intended for the next separation. Recommend how to prepare a solution of the sample (generally a mixture of two or more compounds) for loading onto the column. Describe pros and cons of using too polar of a solvent, too non-polar of a solvent, and too large of an amount of solvent.