Chem 560 – Analysis of dynamic, equilibrating systems

**Titration assignment**

The goal of this assignment is for you to gain experience with determining an equilibrium association constant, Kassoc. using 1H NMR. It will require that you plan, prepare solutions, collect NMR spectra, analyze the resulting chemical shift data to determine Kassoc values. Troubleshooting by changing experimental conditions might be required in order to obtain useful data.

1. You will carry out two single replicate titrations in D2O of the host molecule *para*-sulfonatocalix[4]arene into two small guests, L-leucine and L-phenylalanine. You should set guest concentrations at a fixed value that is somewhere between 2–10 mM, and prepare samples in which you vary host concentrations between 0 and 50 mM. Please collect solid samples and D2O from Alok Shaurya, and plan your solution preparation using known MW values and reasonable volumes of D2O (<25 mL total consumption). You should collect at least 8 concentration data points for each titration, including 0 and 50 mM as two of them. Collect NMR spectra at 300 MHz on the undergrad NMR. Chris Barr (cbarr@uvic.ca) will prepare accounts for you and orient you to the usage of that instrument.
2. Record chemical shift data as a function of host concentration for all signals that vary in chemical shift during the titration.
3. Upload to [www.supramolecular.org](http://www.supramolecular.org). Follow the directions there for how to format your files prior to upload. Use “Bindfit” to fit using the NMR 1:1 host-guest binding models. (Feel free also to try other models if the fits are poor—include other results with your report).
4. Save output data files and prepare figures that show the raw and fitted data.
5. Write a report that includes:
* A point-form summary of your plan and calculations for solution preparation
* Two NMR titration Figures as a stacked plot of NMR spectra (one for each guest)
* Two NMR titration data tables with all chemical shifts vs. concentration
* Two NMR titration Figures as a plot of  vs concentration, with raw and fitted curves and Kassoc values.
* Two NMR titration Figures in which you use a linearized plot to determine Kassoc.
* A Table showing values for Kassoc for each guest, from each method.
* A brief written analysis of the quality of the data and the potential sources for error (≤400 words).

A single pdf file containing all required elements of the report must be emailed to fhof@uvic.ca by June 30, 2017.