# Kinetic Profiling via NMR

NMR can be an excellent tool to follow the kinetics of an irreversible reaction through disappearance of starting material peaks or appearance of product peaks over the course of minutes of hours. You can acquire a spectrum at specific intervals and calculate the rate constant from changes in peak intensities. This is a classical kinetic method, obtaining data on the laboratory time scale. This guide outlines how to obtain kinetic (time course) data, so you can monitor your reactions as a function of time in the NMR tube and determine kinetic parameters. Additionally, this handout assumes you are running a 1H kinetics experiment; for other nuclei, you will need to change the acquisition parameters accordingly.
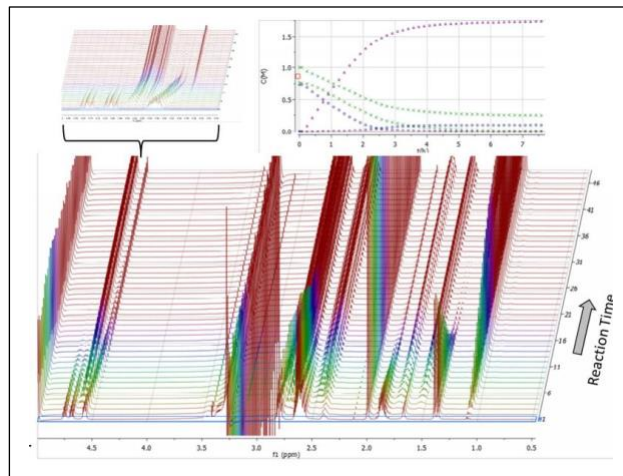
Some Notes Regarding Kinetics

1. If you want your kinetics run to be quantitative, you will need to follow the steps for a quantitative NMR, i.e.,
   - Internal Standard
   - Accurate 90 degree pulse calibration
   - zg pulse sequence (instead of zg30!)
   - *d1 > 5x T_1* relaxation time. As a general rule, 30 seconds is usually enough time for complete relaxation between scans. See the Quantitative NMR handout for more information.
2. Kinetics are very sensitive to temperature. Use the **edte** menu to set the temperature. Make sure not to go above the boiling point or below the freezing point of your solvent!

Data Acquisition

1) You'll want to acquire data of your starting materials *before* you run the reaction, to know where the starting material resonances appear in the spectrum with the same solvent and under the same conditions (e.g., temperature). Follow the normal steps: insert your sample, lock, tune the probe, shim, set the gain, acquire data, etc.

2) Increment the experiment number with **iexpno** for your reaction mixture. Insert your sample. If the reaction is slow, you may have time to quickly shim your reaction mixture before you acquire data. If you are using the same solvent, you can skip tuning the probe. Acquire your first spectrum, making note of the time (the command **dpa** displays the exact time of each acquisition, which you can use to generate the time course profile).

3) After you run the first reaction spectrum, increase the experiment number with. **lexpno.** Use the command **multi_zgvd** and answer the following questions:
   - **Enter fixed delay (in seconds)** (this is how long you want to wait between time points)

- **Number of Experiments** (how many time points you want to acquire)

For example, if you want to acquire 12 time points spaced apart by 5 minutes, you would answer 300 to the first question and 12 for the second. TopSpin will tell you how long the kinetic run will take.

4) Data processing can be done in either TopSpin or Mnova. UO has a site license for the MNova Reaction Monitoring plugin; if you haven't already installed it, you can download from the hotwax server at: http://hotwax.uoregon.edu/mnova.