Kinetics Experiment on AV-500

(modified from University of Oxford instructions)

1. First lock, tune, shim and run initial spectra of your starting materials **before** the reaction to make sure you know exactly where they appear in the spectrum with the same solvent that you use in the reaction under the same conditions, e.g. temperature, preferably on the same day.

2. Having optimized the spectra of your starting materials alone (tuning the probe and shimming), quickly run the first spectrum of your reaction after mixing noting the time you started (the command **dpa** will display the exact end time of each acquisition and these times can be used in generating time course profiles). If your reaction is not too fast, you may have time to do some quick shimming beforehand; re-tuning is not necessarily required.

3a. (option for fixed delays between spectra)
Having run the first reaction spectrum, increment experiment number (**iexpno)**, and use the command **multi_zgvd** *(answer the following questions).*

Choose the "f" option for fixed delay.
Enter fixed delay (in seconds)
e.g. **600** (10 minutes for spectrometer to wait until next acquisition)
Number of Experiments
e.g **30**
The program should tell you how long your kinetic run should take.

3b. (option for variable delays between spectra)
For variable delays between spectra, you first need to create a list of those delays.

Type **edlist vd** to see a list of files.
Select "vdlist1" and **[Edit]** and edit this file in the text window that opens up.
Select **[File]** - **[Save]** the edited file under the same name (vdlist1).
(You should check this file each time as someone else may have edited it for their use since the last time you ran kinetics.)

Having run the first reaction spectrum, increment experiment number (**iexpno),** and use the command **multi_zgvd**

Choose the "v" option for variable delay.
The *Number of Experiments* should match the number of lines in your vdlist1

Processing kinetics data using TopSpin

Please note that kinetics data isn't synced automatically with the datastation, so you will need to sync it yourself. In a terminal window, type:

rsync –aWvx –u av500:/opt/data/username/ /opt/data/username/

4. Process your first spectrum using **efp** and *phase* it perfectly, manually. You need all subsequent spectra in the kinetic run to be phased in exactly the same way.
Type **multicmd**

Specify NORMAL
Enter number of experiments to which to apply each command
*Enter first processing command (e.g., **efp**) – **[OK]***
*Enter second processing command (e.g., **abs**) – **[OK]***
When you have entered all the commands you want to use, select **[OK]**, not **[Close]** instead of entering another command. Then select **[OK]** again to execute the command(s).

Commands you might want to use: **efp** and **abs**. The macro will then use **[efp]** then **[abs]** for all the datasets you have specified; it will phase the specified spectra exactly the same as the first one you phased manually.

5. **Integrate** a good example spectrum of the kinetic run, where you have **both** starting material and product and **save** regions to *'intrng'* using the  icon.
Then write a new integral file with command **wmisc**. Select **[Write New]** and enter a filename *xxx.*

6. Finally, go to the first spectrum of the kinetics run and use the command **multi_integ3** and answer the following:

Use EXPNOs(0) or PROCNOs((1)
0
Enter first experiment No
e.g. 2
Enter no of experiments
e.g. 30
Enter name of intrng file
xxx (from step 5)

In this example, the program calculates all the integrals in files 2…….31 and the results are conveniently presented as a txt file in the experiment you performed the **multi_integ3** command in, with the processed spectrum: /opt/data/*username*/*filename*/*expno*/pdata/*procno*/*filename*_int.txt (the text in italics will vary depending on the user and filename).

This table of integral values can easily be imported into **Excel** as a Space and Tab delimited file for plotting and for calculations of rate constants.