

DRX 400 Training Manual

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Safety

All researchers working in 1232 Hach Hall must complete the EH&S Course "*Fire Safety and Extinguisher Training.*" Please do not prepare samples directly in 1232 Hach Hall. Aprons, safety glasses, and rubber gloves are available in 1238A Hach Hall if you wish to conduct your sample preparation in CIF facilities. Researchers may wear lab coats and safety glasses in the 1232 Hach Hall, but please remove all gloves before handling NMR equipment or computers. Please do not bring any large ferromagnetic objects into the lab without permission. This includes certain chairs, bicycles, gas cylinders and tools.

Properly dispose of waste solvents and glass pipettes in the containers provided in 1238A. There is a broken glass container located in 1232 Hach in case of broken NMR tubes or other glassware. All of the computers in this lab have direct links from the desktop to MSDS sheets, the EH&S Laboratory Safety Manual and to the CIF Safety Manual.

Some safety concerns specific to high-field cryogenic superconducting magnets include:

- Users should remove credit cards, cell phones, mp3 players, keys and other ferromagnetic objects from pockets before approaching a magnet.
- Users with pacemakers or joint replacements should have a staff member assist them with the insertion of samples into the magnet as to prevent serious harm or injury.
- In case of magnet quench, the room will be filled with gaseous helium, which will be evident from a white cloud and an alarm sounding on the oxygen sensor. If you are in the room during a magnet quench, please exit as soon as possible – crawling on the floor if need be to reduce helium inhalation. If you are outside the room during a magnet quench, do not enter the room without proper breathing apparatus until the oxygen sensor alarm has stopped sounding.

Introduction

You must receive training before using this piece of equipment. The Bruker DRX400 features a narrow bore 9.4 tesla/400 MHz magnet equipped with a normal geometry $^2\text{H}/^1\text{H}$ /BB probe tuning to nuclei in the frequency range of ^{15}N - ^{31}P on the broadband channel and a triple resonance inverse probe with $^2\text{H}/^{31}\text{P}/^1\text{H}$ /BB channels. The normal probe is capable of variable temperature experiments in the range of $-150\text{ }^{\circ}\text{C}$ to $+150\text{ }^{\circ}\text{C}$. The inverse probe is capable of variable temperature experiments in the range of $-50\text{ }^{\circ}\text{C}$ to $+50\text{ }^{\circ}\text{C}$.



Overview

The DRX400 is controlled by a Red Hat Linux PC communicating via serial port to the spectrometer and Ethernet to the internet. The computer is a part of a local area network in 1232 Hahc Hall and is safeguarded behind a firewall. In order to minimize instrument time when users are waiting, the data acquired is immediately accessible on one of four data stations in 1232 Hach. Either Topspin or the third party MNova software can be used to process the data. The MNova software is available on the data stations and is generally used for processing, plotting and printing data. The data is also accessible via the [CIF Research Files Cloud Storage](https://cif-research-files-cloud-storage.com/). When you log out, your data is instantly uploaded to the storage cloud. More information regarding Remote Data Access is available on the CIF website: <http://dev.cif.iastate.edu/remote-data-access>

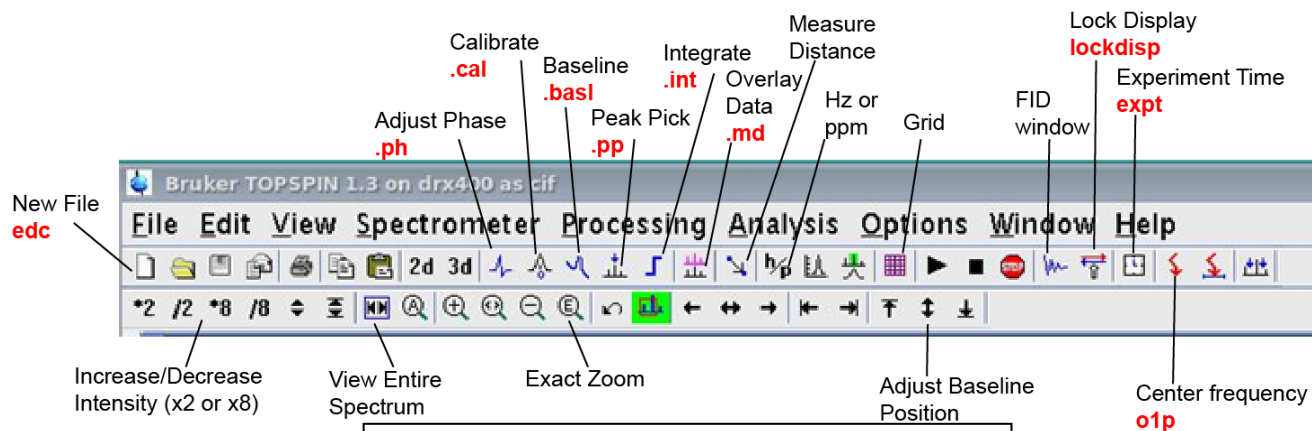
Start up

After logging in, Topspin will start automatically.

1. Be sure the instrument is set up for the desired nucleus.
 - a. For all nuclei except proton, the probe tune values must be the same as those written on the sheet taped to the front of the magnet.
2. Slide the sample into the spinner and adjust the depth using the depth gauge.
 - a. Sample height must be 5 cm (0.7ml). This is required to make shimming faster and improve spectra.
3. Wipe both the sample and the spinner with a Kimwipe.
4. Remove the cap from the top of the magnet. Turn the eject air by pressing the **LIFT ON/OFF** key on the upper left hand corner of the BSMS panel. Once the eject air has come on, place the sample in the top of the magnet.
5. Push the **LIFT ON/OFF** key once more to lower the sample into the probe.

Set up a 1D Experiment

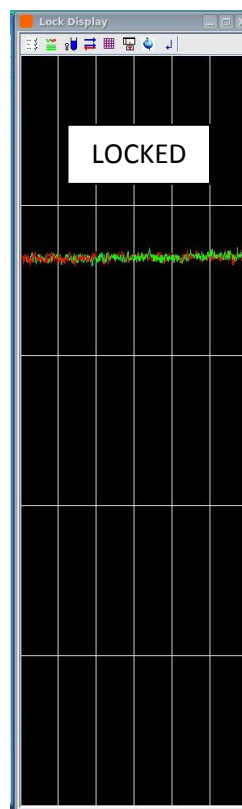
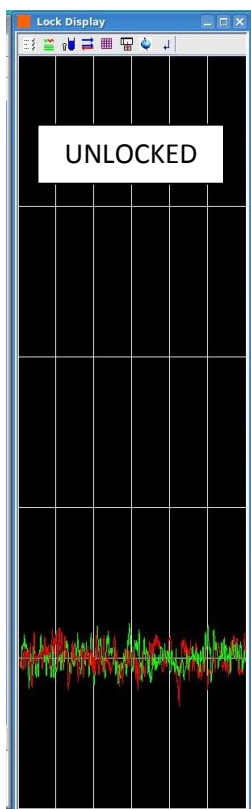
6. Create a new data file by typing **edc <enter>** and enter the desired **NAME** (typically the name of your sample or however you name your files) and **EXPNO** (1, 2, 3....) in the window that appears. And then click **SAVE**.
 - a. Read in a standard file by typing **read <enter>**. Click the desired experiment with left mouse button, and then click **COPY ALL**.
 - b. OPTIONAL: You may type **eda <enter>** to modify the parameters. NS may be modified by typing **ns <enter>** and enter the number and hit ok. Type **ii<enter>** and wait for the **ii:finished** message to be displayed in the bottom left corner of the Topspin window.



Topspin 1.3 Menu Bar – Some buttons also have a corresponding command, shown in red.

Locking and Shimming

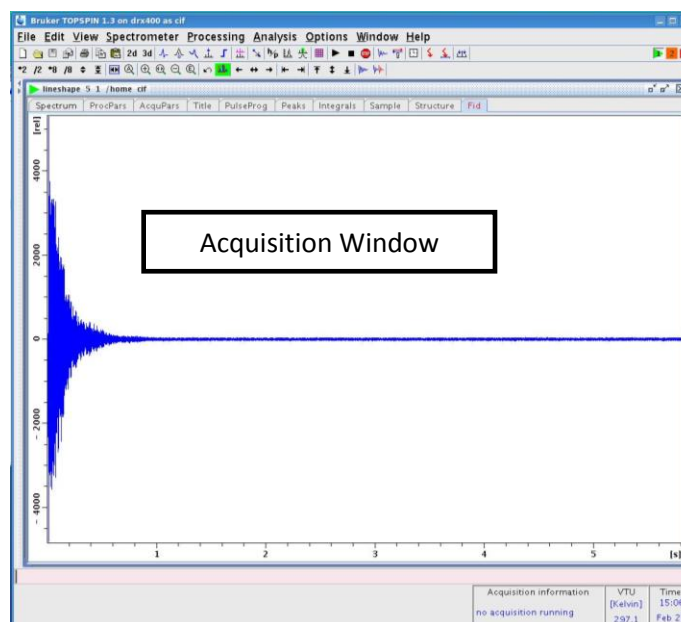
7. If you do not see a lock window type **lockdisp<enter>** in the command line. The lock signal is a green/red line moving across the lock display. Type **lock <enter>** and select the correct solvent for your sample.
8. Using the BSMS panel, adjust **LOCK GAIN** so that the lock signal is between approximately 2/3rds of the way up the lock display screen. Make sure the **FINE** button is lit on the BSMS panel.
9. Type **lock<enter>** and choose your solvent to lock. When the sample is unlocked, the red/green line will appear at the bottom of the **lockdisp** window as shown in the first window below. When it is locking, an FID will appear when the locking finds the correct field setting, and then it will gradually increase to find the correct **LOCK GAIN** setting, as shown in the second image below. Once it is locked, the red/green line should be stable in the upper half of the **lockdisp** window. You can adjust the position of the red/green line (the lock level, essentially) by increasing or decreasing **LOCK GAIN** and **LOCK POWER** on the BSMS panel as shown above.



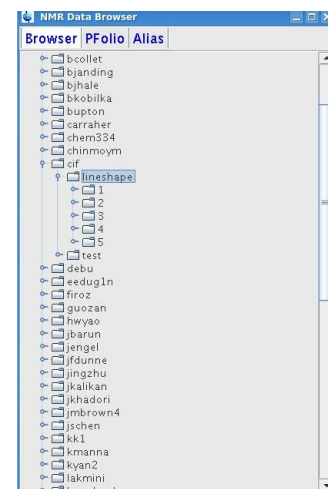
10. Choose a shim by pushing its respective button on the BSMS panel. Adjust the **[Z]** by gradually spinning the wheel until the lock signal is maximized. Choose another shim (typically **[Z2]**). You may reduce the **LOCK GAIN** accordingly to keep the lock signal visible. Typically **[X]** and **[Y]** don't require much adjustment, but you can also adjust these shims for optimal lineshape. When satisfied, push **[STBY]**.

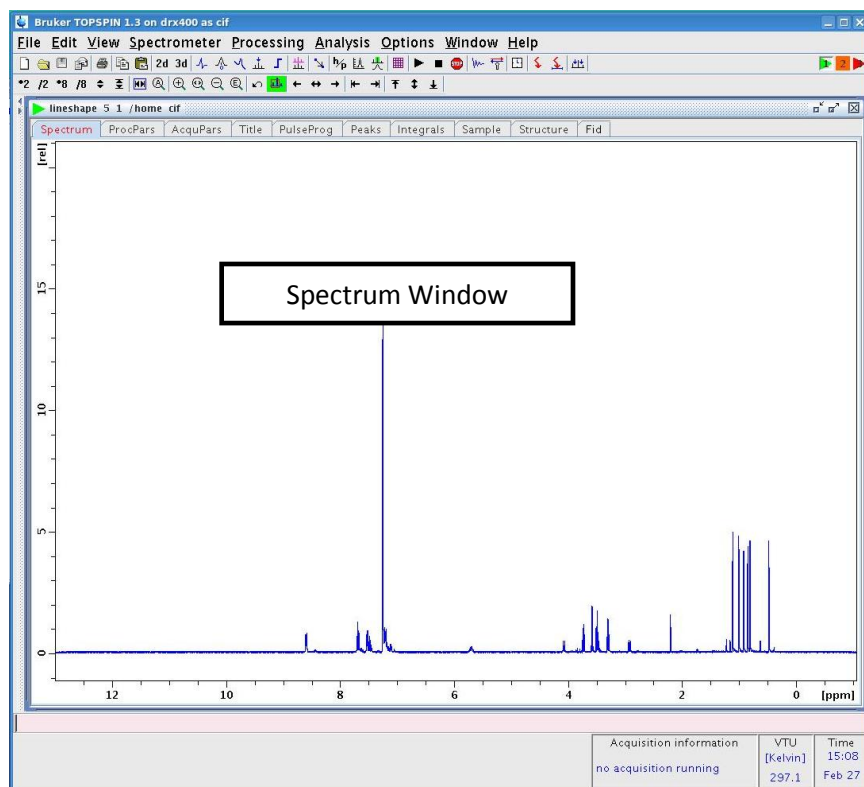
Acquiring Data

11. Type **rga** <enter> in the command line. Wait for the “rga: finished” message to be displayed.
12. Optional: Type **expt** <enter> to make the software calculate the length of the experiment.
13. Type **zg** <enter> to start the acquisition.
14. The software will automatically switch to the acquisition window after typing zg, and should show the FID (free induction decay):



15. To halt the acquisition, type **halt** <enter> will stop the experiment at the current scan.
16. To view the spectrum during acquisition, type **tr**<enter>
 - a. Then after you see “checklockshift: finished” in the lower left corner, type **ef; apk**<enter>
17. Type **ef; apk**<enter> to Fourier transform and auto-phase the data. After **apk** has been typed once, subsequent Fourier transforms can use **efp** (uses the phase found in **apk**).
18. Data in Topspin is auto-saved. Once the acquisition is finished, no further actions are required. All of your previous data can be found in the data browser (See Photo →).



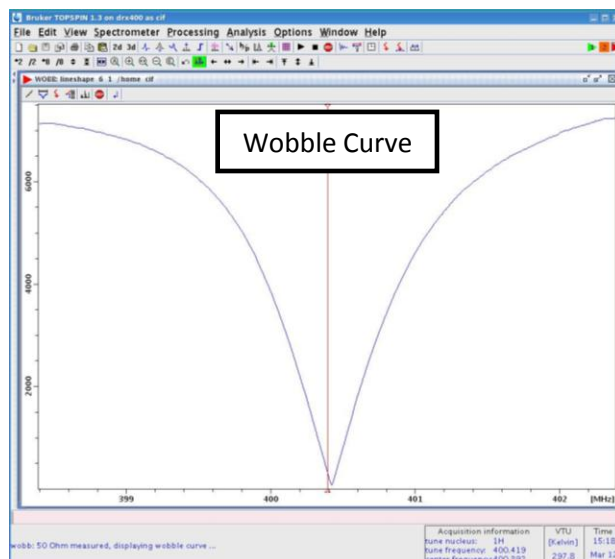


Setting up a 2-D Experiment

1. See the **Additional Experiments** section below for what types of 2D's are available.
2. Collect a 1D proton spectrum and process the data as directed above. Integrate the region of the ^1H spectrum that you wish to use for the 2D acquisition.
3. Type **edc <enter>** to create the 2D file. Enter NAME and for your 2D file in the window that appears. Click **Save**.
4. Type **edc2 <enter>**. Enter **NAME2** and **EXPNO2** of your ORIGINAL proton file. Click **Save**.
5. Type **read <enter>**. A complete list of available experiments will be displayed. Be sure to select a 2D experiment such as COSY, TOCSY or HMBC. Select the experiment you want and then click **Copy All** and **OK**.
6. Type **solvent <enter>** and select the solvent.
7. Type **ii <enter>** and wait for ii:finished to be displayed
8. Type **xaue <enter>** to collect the data.
9. To look at the spectrum during acquisition, type **xfb <enter>**. The 2D spectrum can be viewed after at least 2 slices have finished acquiring. The **tr** command does not work during 2D acquisitions.

Tuning the Probe

1. For nuclei other than ^1H , the X channel of the probe is tuned using the tuning wands under the probe. Acceptable tuning can be obtained by selecting the standard values. The standard values are taped to the front of the magnet (there is a different set for the TBI and the Normal probes).
2. Fine tuning the probe while your sample is in the magnet may increase S/N.
3. To fine tune the probe:
 - a. Have your sample inserted in the magnet. Lock and shim the sample.
 - b. Do **edc<enter>** to create a new file as directed above.
 - c. Type **read<enter>** or **rpar<enter>** and load the experiment of choice.
 - d. Type **ii<enter>**
 - e. Type **acqu<enter>**
 - f. Type **wobb<enter>** to display the wobble curve. The lowest frequency nucleus will wobble first, which will always be the X nucleus that you are tuning.
 - g. Adjust the tuning wands to the numbers listed on the standard values table. Once you have entered the **Tune** and **Match** values on the standard values table, adjust the last digit of the **Tune** and **Match** values until the wobble curve dip is centered and lowered to the baseline on the screen.
4. When the wobble curve is adequately centered and minimized, type **halt<enter>**
5. Proceed with acquisition as above.



Ejecting Sample & Exiting

1. Push the **LIFT ON/OFF** key to eject the sample into the probe.
2. Remove the sample from the magnet and return the spinner to the depth gauge.
3. Push the **LIFT ON/OFF** key to turn off the eject air.
4. In the command line, type **exit<enter>** and then click **OK**.
5. Place the cap on top of the magnet and logout of the computer by right-clicking on the desktop.

Additional Experiments

Variable Temperature Experiments

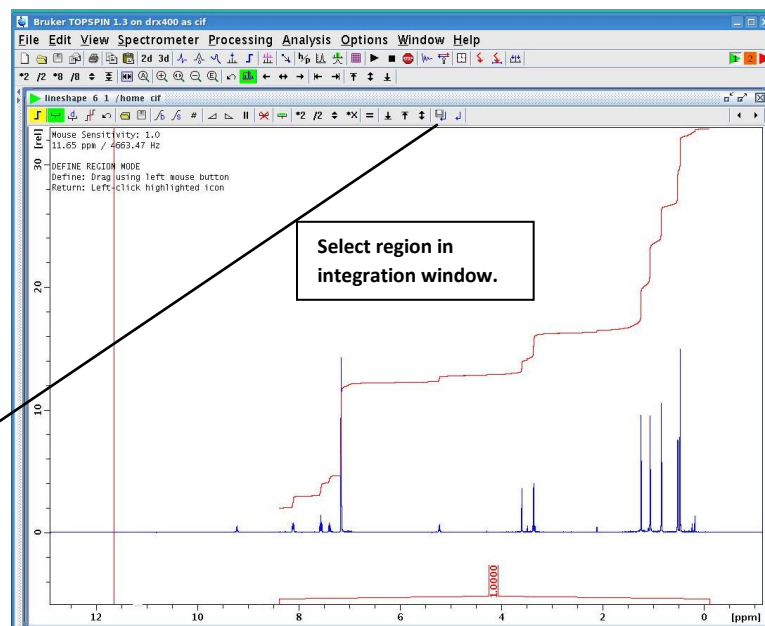


1. Temperature range of the **Normal Probe**: -150 °C to +150 °C
2. Temperature range of **the Inverse Probe**: -50 °C to +50 °C
3. Be sure the **GAS FLOW** is greater than 5 on the BVT Unit.
4. Press the **HEATER** button, read means it is ON.
5. Setting the temperature of the BVT-2000
 - a. Type **te<enter>**
 - b. Enter the temperature in **Kelvin** and hit ok.
 - c. Type **teset<enter>**
6. For low temperature, start the FTS Air Jet unit (Photo->)
 - a. Turn on the power on the top then the bottom unit.
 - b. Press **SP4** on the top unit.
 - c. Wait for the top unit to read -70 °C before setting another temperature
 - i. SP4 = -88 °C
 - ii. SP3 = -30 °C
 - iii. SP2 = -10 °C
 - iv. SP1 = -0 °C
7. The temperature on the BVT-2000 is the probe temperature. Increase the **GAS FLOW** to reach lower temperatures, but increasing the gas flow too much may lift your sample.
8. When finished:
 - a. Turn **OFF** the **HEATER** , making sure the red light is off.
 - b. Turn the **POWER** off on the FTS Unit, first bottom then top.
 - c. Return **GAS FLOW** to 5 if it had been changed.



Kinetics Experiments

1. Collect a 1D ^1H spectrum and process the data as directed previously.
2. Integrate the region desired for kinetics experiment:
 - a. Enter the integration window by typing `.int<enter>`
 - b. Click and drag the cursor over the desired region.
 - c. Hit the “Save & Return” icon or type `.sret<enter>`



3. Type `edc<enter>` to create the 2D filename. Enter the **NAME** and **EXPNO** for your kinetics file in the window that appears.
 - a. Once the new data window opens, type `edc2<enter>` and enter the **NAME** and **EXPNO** of the 1D ^1H that was acquired in step 1. This step “points” to the previous data set and the selection of the region of interest. Click **SAVE**.
4. Type `read<enter>` and select **kinetics**. Click **COPY ALL** and **OK**.
5. Type `solvent<enter>` and enter the solvent name.
6. Type `edlist vd<enter>` and enter the desired delays. The delays start after the first spectrum, and they are listed in seconds (i.e. 3600 seconds = 1 hour).
7. Type `ns` to enter the desired number of scans (usually 1 scan per time point).
8. Type `1 td<enter>` and enter the desired number of experiments (the **vd list** can contain more points than you enter here).
9. Type `1 si<enter>` and enter the next power of 2 greater than the **1 td** value entered in the previous step. (i.e. if you enter **1 td=15**, then **1 si=16** If **1 td=24** then **1 si=32**)
10. Type `ii<enter>` and wait for **ii: finished** to display in the lower left corner.
11. Type `xaua<enter>` to start the acquisition. Process data with `xf2<enter>`

Kinetics on X (other than ^1H) Nucleus

1. Acquire a 1D X nucleus experiment as above for ^1H .
2. After completing step 3 above, type `pulprog kinetics<enter>` for no decoupling or `pulprog kineticspg<enter>` if you want ^1H decoupling.
3. Complete steps 5-9 as above.
4. Type `aq_mod<enter>` and choose **qsim**
5. Type `aunm auget1d<enter>`
6. Type `ii<enter>` and wait for **ii: finished** to display in the lower left corner.
7. Type `xaua<enter>` to start the acquisition. Process data with `xf2<enter>`

T1 Experiment

1. Collect a 1D ^1H spectrum and process the data as directed previously.
2. Narrow the window and determine the 90° pulse.
3. Type **pulprog t1ir<enter>**
4. Type **parmode 2d<enter>**
5. Type **vdlist VDLIST<enter>**
6. Type **edlist vd<enter>** and enter the desired delays. The delays start after the first spectrum, and they are listed in seconds (i.e. 3600 seconds = 1 hour).
7. Type **l4** and enter the number of delays in the list.
8. Type **zg** to start the experiment.
9. To process the data, type **xf2<enter>** and phase correct if needed (enter the phase correction window by typing **.ph**)
10. Type **rspc<enter>** and then **basl<enter>**
11. Type **def-pts**
12. Middle mouse button on the peaks, left mouse, save & return.
13. **t1/t2**
14. **pd**



Bruker DRX400

Experiment Guide

Common Topspin Commands

- **absn** – auto baseline correction
- **apk** – auto phase correction
- **ased** – all experimental parameters (pulse lengths, number of scans, delays, etc.)
- **.cal** – interactive calibration window
- **d1** – recycle delay, time in between scans
- **edc** – create new experiment from a parameter set
- **ef** – fourier transform and apply exponential broadening, controlled by **lb**
- **efp** – fourier transform, exponential broadening and phase (either phased with **apk** or **.ph**)
- **ej/ij** – eject air/insert air
- **go** – continue acquisition after it has ended (add more scans)
- **halt** – abort acquisition
- **humpcal** – peak width calculation
- **lb** – line broadening, typically <1.0 Hz for ^1H , 3-5 Hz for ^{13}C
- **lock** – lock on a solvent, choose from a popup window
- **lockdisp** – display lock window
- **ns** – number of scans
- **o1p** – center of spectrum in ppm (**sw** controls spectral window size)
- **.ph** – interactive phase correction window
- **.pp** – interactive peak picking window
- **rg** – receiver gain, set automatically with **rga**
- **rga** – automatic receiver gain setting
- **rpar** or **read** – read parameter set
- **rsh** – read shim file, **rsh standard.shim** to load standard shim set
- **.sino** – signal to noise interactive window
- **.sret** – save and return from peak pick/integration/phase window
- **sw** – spectral window size in ppm
- **tr** – transfer data during acquisition, follow with **ef**; **apk**
- **wsh** – write shim set specifically for your samples – save with your initials
- **wrpa** – copy expt parameters & acquisition to another file
- **xaua** – start a 2D and call the spectral window from the **edc2** setting
- **xfb** – fourier transform a 2D such as COSY or HMBC
- **xf2** – fourier transform an arrayed 2D such as kinetics or T1
- **zg** – zero, go – deletes any old data and starts acquisition. Acquisition can be continued with **go**