Anasazi EFT60 Nuclear Magnetic Resonance Spectrometer

Solution height must be 4.0 cm (0.7 mL in tube).

Remove previous sample by opening top door (lift front edge), holding in the *launch* button and catching tube. (Be ready when the tube pops up!) Place tube in spinner, and adjust the height using front gauge. Place spinner and tube in sample shaft and use your thumb to briefly seal the shaft. Use the flashlight to check if the lowered tube is spinning.

Sign the logbook. (Always note any difficulties to alert others to potential problems.)

The Anasazi NMR uses two programs, one to collect data (PNMR) and one to analyze data (NUTS). Usually PNMR is the left window and NUTS is the right window. The advantage of the two programs is that you can examine collected data without interrupting ongoing data collection. Unfortunately, the PNMR program requires you to press *[enter/return]* to accept a value and the NUTS program uses *[enter/return]* to cancel the command.

zg [enter] in PNMR (left window at bottom left) to **collect dat**a.

Type file name or preferably use the default (pnmrfid) by pressing *[enter]*.

Wait for data collection to finish, then switch to the NUTS program (right window).

a0 in NUTS to **import and Fourier transform** data (*ga bc ft qp fb l ^m ^f*).

Type file name or preferably use default (pnmrfid) by pressing *[enter]*.

While holding mouse down over known peak, *sz* [value] *[enter]* to set ppm.

Use mouse wheel to adjust peak heights so tallest sample peak reaches the top.

To zoom in, type *zo* or double click the left mouse button. The cursor will change to *+zo*. Drag to select new width. Click the right mouse button. *[enter]* to exit the zoom mode and change cursor to an arrow. CTRL-F for full width, CTRL-E to rezoom.

To phase use *qp* or *ap* (no enter) or *ph* (horizontal mouse drag) then *[enter]*.

To label or update peak positions use *pp*; *pf* turns labels off; *mh* is the minimum height for labeling; *dp* manually labels peaks.

To see the FID, use *ga* instead of *a0*.

a8 (no enter) to **integrate** (*ai id z*)

Double click to left of peak and single left click to right of peak to add new region.

Use *c* to clear all regions and start again.

Click within a region then *v* [integer] *[enter]* to set the region integral value.

Drag far left slider to adjust integral scale.

Drag near left slider to adjust vertical zero.

[enter] [enter] to leave integral mode. Use CTRL-I to turn integrals off.

a9 (no enter) to **print** (*zo f ^m ^e pl*).

Normal response is *12 tab -.5 [enter]* for proton or *250 tab -10 [enter]* for carbon.

Inset plots: *zo* and drag to select region, CTRL-E (or right click) to expand, *[enter]*. *mo* to inset, click *A* to add view, drag, then CTRL-F (or right click) to resume full scale. Can *mo* again to position or remove box. *Delete inset* for every new sample.
The previous page assumes nucleus (*nu H1* or *nu C13*) *fo* and *shim* are set. See below.

Acquisition parameters (PNMR software). Use [enter] after a command to get a dialog box asking for the value, or use command space value to skip the dialog box.

Nucleus *nu [enter] H1* or *C13*
(Choose proton or carbon-13; the nucleus will be displayed as the command prompt.)

Size *si [enter] 16k* (8 sec) or *8k* (4 sec) or *4k* (2 sec) or *2k* (1 sec)
(To set the acquisition time, you change the data size *si*. *8k* is normal.)

Number of scans *ns [enter]* value
(Reasonable values for proton are 1, 4, 8 or 16 and for carbon are 64, 128, 256. For more scans see *bapr* below.)

Relaxation delay *rd [enter]* value
(seconds. Shorter relaxation delays require smaller pulse widths. 2 is reasonable.)

Pulse width *pw [enter] 17* or less
(μseconds. Use the 90° pulse, which can be measured with *h90cal*, or less.)

Receiver gain *gs [enter]*
(This displays the FID signal, which should not reach the red horizontal lines. Press CTRL-G to get a dialog box and type a new value, 1-100. For protons in water the correct gain is about 5; for a typical organic molecule dissolved in chloroform the gain is about 30. In this mode you can also change the pulse width using CTRL-P and the relaxation delay in seconds using CTRL-R. When you are finished use CTRL-Q to quit the adjustment.)

Field Offset *fo [enter]*
(Run a spectrum, go to NUTS, find position of known peak, go to PNMR, *fo [enter]* value for found and known position.)

Collect data *zg [enter]*
(Erases previous data file, starts acquisition, and saves new data. To collect more scans without erasing, for example a second set of 256 scans for carbon-13, use *go [enter]* instead of *zg*. CTRL-S to stop.)
zggd does gated decoupling (carbon NMR proton coupled with NOE.)

*B*lock *a*veraging with *p*eak *r*egistration (for longer runs than *ns* 256, instead of *zg*)
ns value *bapr [enter] [enter] 24 [enter] [enter]*
Uses default filename, my_bapr, and 24 blocks of data. Go to NUTS, *a1, zo* to select a single strong isolated peak *0 [enter]*, CTRL-F12 to select my_bapr and process. Can examine while still collecting data. *lb 1*

Write/Read file *wr [enter]* or *re [enter]*
(Write/Read the FID data file, current parameters to a .ini file, or current shim settings to a .shm file.)

Other commands: COSY (proton-proton correlation), INVREC (measure T1), HET (proton-carbon correlation), DEPT135 (CH and CH_3 up, CH_2 down, quaternary C absent)

Major tuning maintenance shimming with a water sample: *suprep [enter]*
dtool [enter] (values of all shim settings)
shim2 [enter] (water sample) for width and asymmetry near baseline, once a semester
shim3 [enter] (relaxation delay 3) for resolution and line shape. CTRL-K if looks good.

Fine tuning sample, CTRL-S toggles FID and spectrum during *zg*.
prep [enter] (water sample) done weekly
shim [enter] (shim only, not autozero) for sample if needed