BRG Guideline: Basic NMR Spectrometer Operation

The following guidelines provide an overview to operation of the departmental Bruker NMR spectrometers and are to be used as a memory aid for new users. Before using NMR spectrometers for the first time, users **must** attend a training session. Rodger Kohnert should be consulted immediately if any problems are encountered when using the instruments.

1. Arrival at NMR

- log-in and run Topspin software (single click on icon)
- press "lift" button to engage sample air lift
- place NMR sample tube in spinner and adjust to correct depth with gauge
- gently place NMR tube/spinner on air cushion in center of magnet
- turn-off air lift (press "lift" button again), and engage "spin" button
- sample will sink into the magnet, and spinning will start

2. Data acquisition **[summary: new → zz → ns → lock → rga → zg]**

- **`new <CR>`** set-up new file for NMR experiments connected to this sample (nb. more than one experiment can be recording under same file name using different exp. no. extensions)

  filename: use your lab book ref. plus an appropriate extension letter to indicate origin of sample, e.g. "**`PRB502A`**"

  exp. no.: distinguishes between different NMR experiments run on the same sample; it is recommended that you standardize how this index number is used, e.g. 1 for ^{1}H NMR, 2 for ^{13}C NMR, 3 for DEPT135, 4 for COSY

  proc. no.: 1

  solvent: choose solvent from drop-down list as appropriate

  title: use the following format: sample ref., approx. amount, # of scans, solvent, NMR field strength, e.g.

  "**`PRB502A 5 mg 16 sc CDCl3 300 MHz`**"

- **`zz <CR>`** a macro which brings up a list of common NMR experiments; choose as appropriate (nb. protonstd 14 or 16 refers to desired spectral width (in ppm), use smaller width useless you anticipate unusually downfield signals)
- **`ns <CR>`** set number of scans as appropriate; for standard ^{1}H NMR's and samples of ca 5 mg, 16 scans is ample (nb. make sure ns is a multiple of 4)
- **`lock <CR>`** automatically sets deuterium lock; choose appropriate solvent from list; machine will take approx. 5 seconds to find and set the lock and indicates when finished

shimming: while viewing lock display window, adjust "z" and "z^2" (on axis) to optimize shim set (i.e. make lock line as high as possible); do z first, then z^2, then go back to z etc. (nb. activate "fine" control as needed); if line goes off screen reduce "lock gain" (can use this function to raise and lower line as desired); when happy with shim, press "std. by" to prevent accidental change of parameters

- **`rga <CR>`** automatically set receiver gain; machine will pulse sample with rf and monitor strength of NMR signal; procedure takes 5-10 seconds to complete
- **`zg <CR>`** "zero go"; command starts data acquisition and will accumulate FID for the desired number of scans

  to stop data acquisition early, type "**`halt <CR>`**", this stops scans *and* saves FID data; *nb. the alternative stop command,* "`stop <CR>`" *does not save collected data to the hard disk*

3. Data processing

[summary: ft → phase → baseline → calibrate → integrate → pick peak]

- **`ft <CR>`** to perform Fourier transform on collected FID data; zero-filling can be conducted prior to FT by typing "**`si <CR>`**" and then doubling number (e.g. from 32 K to 64 K), this operation is optional and improves the cosmetic appearance of peaks making them smoother
- enter phase correction by clicking on icon (or access from processing menu)

  zero-order: hold mouse pointer over "0" icon and with left mouse button depressed, move mouse up/down to change zero-order phase angle; adjust until line shape of largest peak in spectrum is nicely symmetrical about its base

  first-order: hold mouse pointer over "1" icon and with left mouse button depressed, move mouse up/down to change first-order phase angle; adjust so that a peak distant from the largest one has good symmetrical shape about its base
- **`abs n <CR>`** performs automatic baseline correction without auto-integration
- zoom in on desired ref. peak, click the calibrate icon, move calibration line onto the peak and click left mouse button; enter the desired ppm value (see BRG website for values; nb. $\delta_{H\ (CHCl3)} = 7.26$ ppm)
- click integration icon; integrate by dragging integrals across desired regions; click save and exit icon when happy with integrals
- pick-peak by dragging box across all signals of interest; click save and exit icon when happy with peak-picks
- **`plot <CR>`** enter NMR plot software and call-up a BRG standard plot template